PETROLEUM PROCESSING

PUBL
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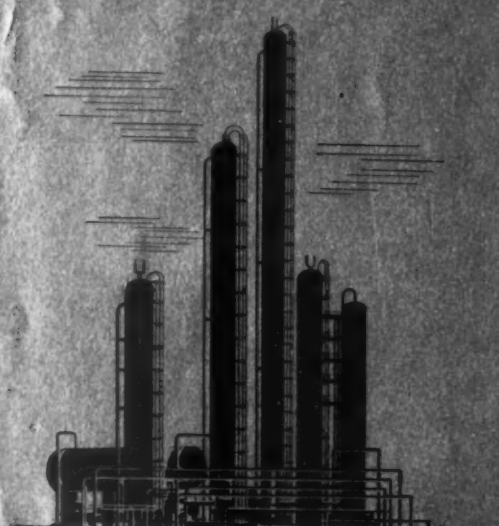
VOLUME 2, NUMBER 11 NOVEMBER, 1947

Making Catalyst for Testing At Riverside (page 834)

FORMERLY MONTHLY TECHNICAL SECTION OF NATIONAL PETROLEUM NEWS



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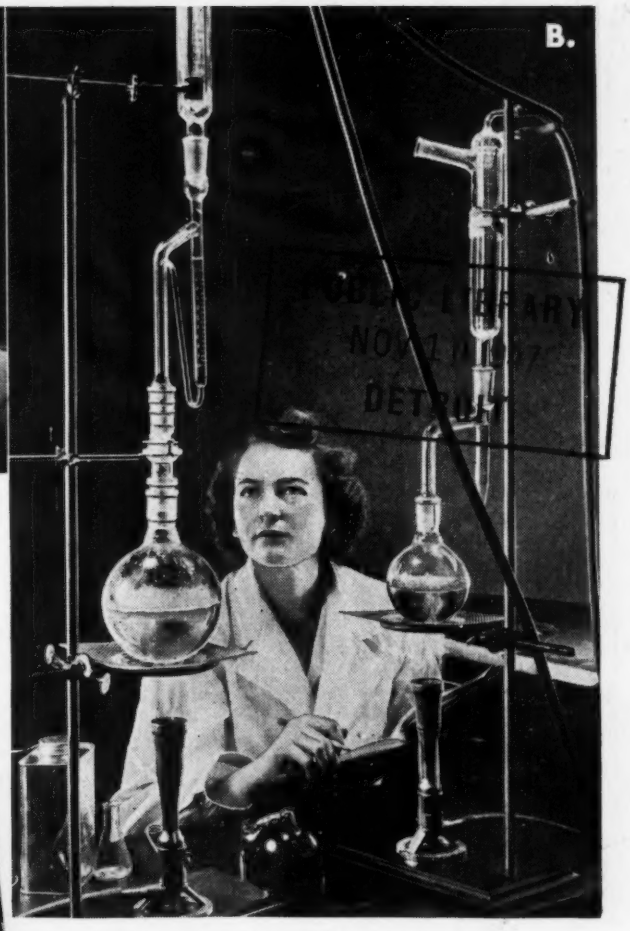
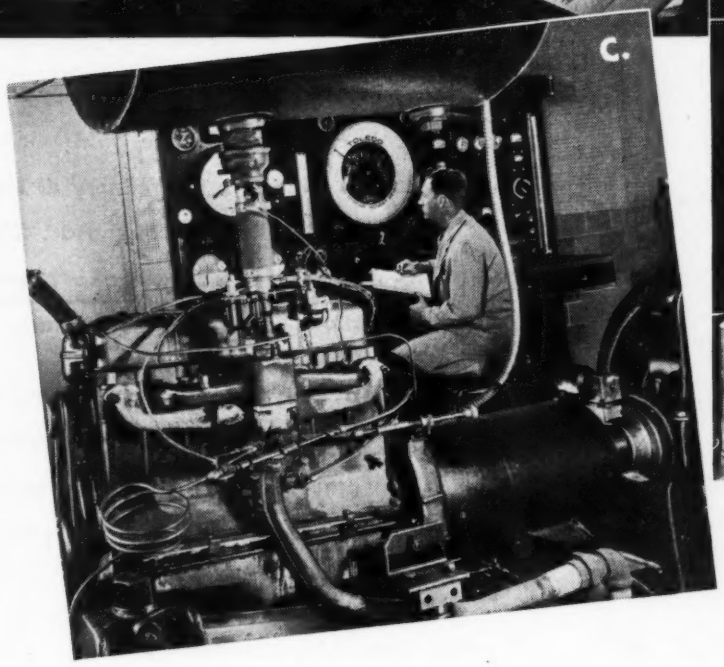
THE HUDSON organization, qualified by years of experience in the successful design, construction and operation of major hydrocarbon processing plants, offers its specialized services for projects in any part of the world.





A. Spectroscopic tests efficiently determine the presence of non-petroleum components of oil samples.

B. Checking a used lubricant for dilution by water or gasoline.

C. The performance of engines using different combinations of gasoline and oil is measured under many different conditions.

Checking

ON THE PRIVATE LIFE OF ✓ ✓ GASOLINE AND OIL

Modern, high-speed engines require lubricants which will withstand high temperatures and pressures—but which will not produce undesirable chemical reactions when used with modern gasoline and antiknock fluid. If gasoline and oil are not compatible under engine operating conditions, a wide variety of complex troubles can result.

Laboratory inspection tests on fuels and oils give much valuable data; however, there is no substitute for actual engine operation in determining the performance in actual service to be expected from a specific combination of gasoline and oil. The study of the reactions which occur in engines when different gasolines and oils are used forms an important part of Ethyl research.

In cooperative programs with oil companies, the Ethyl Corporation has operated single-cylinder and multi-cylinder engines on dynamometers for thousands of hours, and has operated vehicles on the road for hundreds of thousands of miles, in order to study the compatibility of different types of oils and gasolines. Data from these programs are of great value to the cooperating companies in indicating fuel and oil combinations which are conducive to engine cleanliness and durability.

Utilizing its more than twenty years of experience and close association with the technologists of the oil and automotive industries, Ethyl Corporation's research and service organization cooperates with oil companies, both in the investigation of current problems and in the development of fuels and lubricants for the future.

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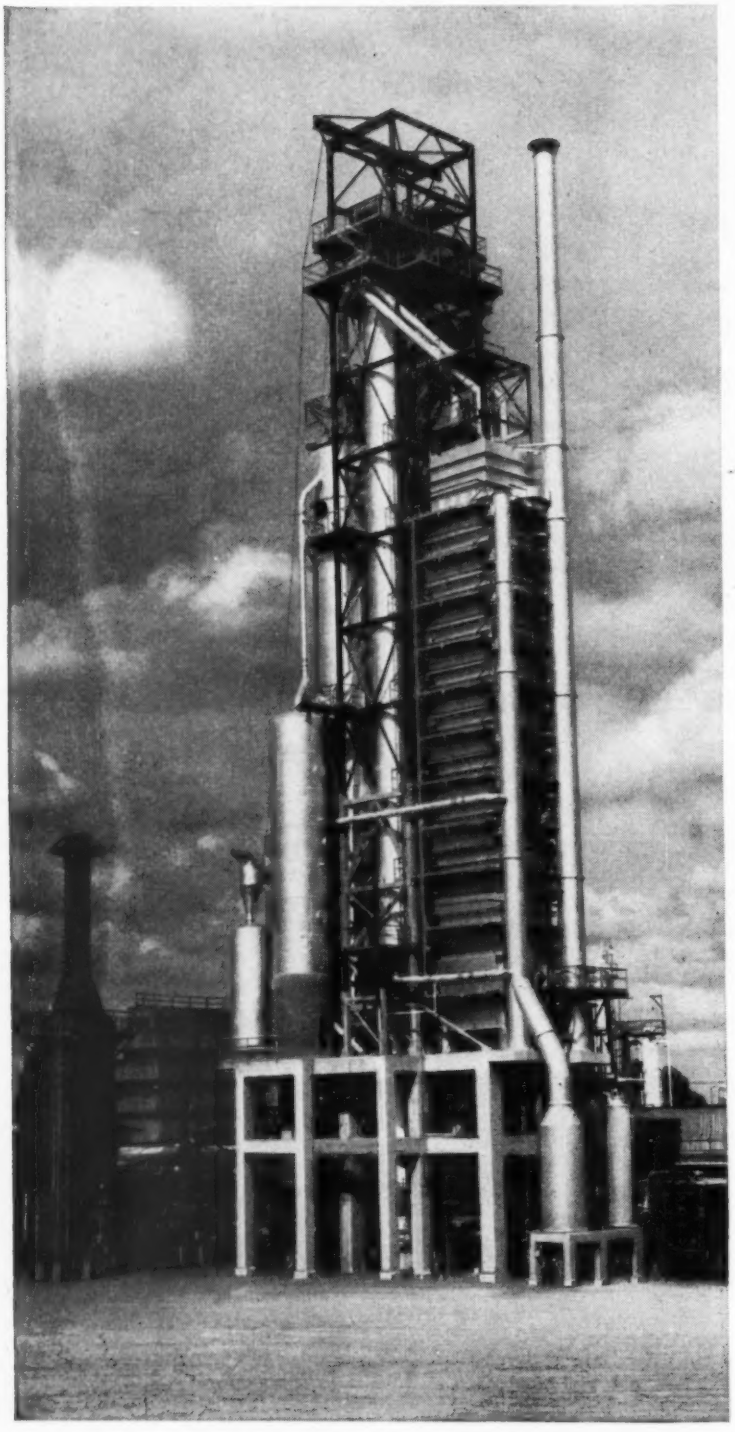
Lion's new TCC unit goes on stream!



On September 16, the sixty-seventh Houdry-licensed cat cracker—and the thirty-eighth T C C—went on stream in the refinery of Lion Oil Company at El Dorado, Arkansas. This unit is of 4,500 bbl. fresh feed capacity, with 900 bbls. additional capacity for recycling.

This new Lion unit will process high-sulfur crude (1.8% in the reactor charge). Corrosion protection is provided by lining the reactor, fractionating tower and all vessels with alloyed steel.

In actual operation, this new T C C unit is running 20% over design capacity, enabling Lion to maintain the high octane quality of its motor gasoline output at considerably reduced costs. And, with built-in recycling, the refiner will be able to vary his product distribution at will, according to seasonal demands.

These are advantages inherent in T C C which have made it the catalytic cracking process outstandingly favored by refineries of relatively moderate size. Details will be furnished gladly to interested refiners.

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VOLUME 2
NUMBER 11
NOVEMBER, 1947
PUBLISHED
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PETROLEUM PROCESSING

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OILGRAM NEWS
OILGRAM PRICES
PLATT'S OIL PRICE HANDBOOK

Covering the Management, Design, Operation, Equipment, Products of Plants Processing Crude Oil and Natural Gas into Fuels, Lubricants, Chemicals

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for November, 1947

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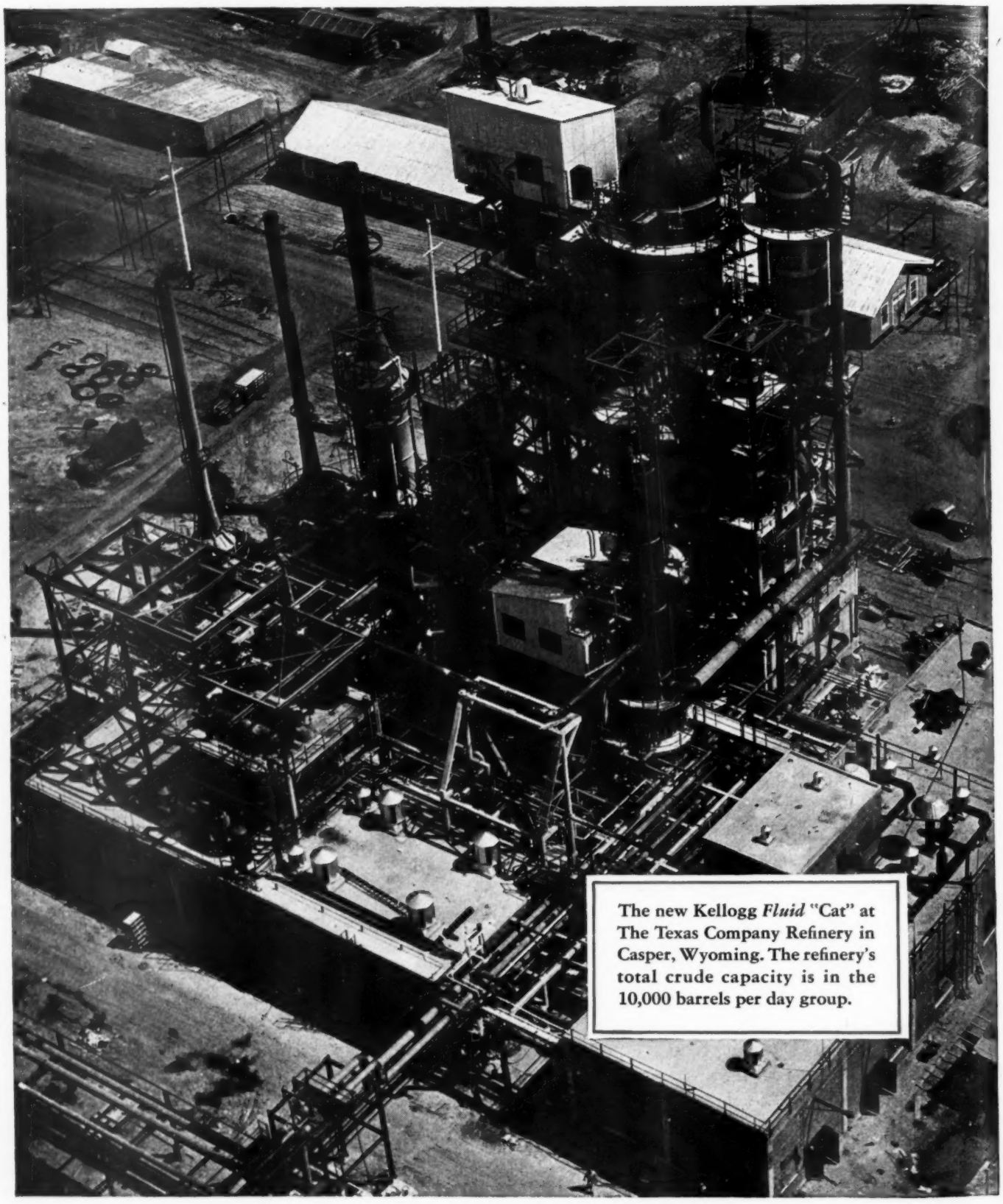
CHECK THE RECORD



The new Kellogg *Fluid* "Cat" at The Texas Company Refinery in Casper, Wyoming. The refinery's total crude capacity is in the 10,000 barrels per day group.

ON KELLOGG FLUID "CAT" CRACKING

Upon completion of existing contracts, the total fresh feed charged to Kellogg-built Fluid "Cats" will run close to 700,000 barrels per day!

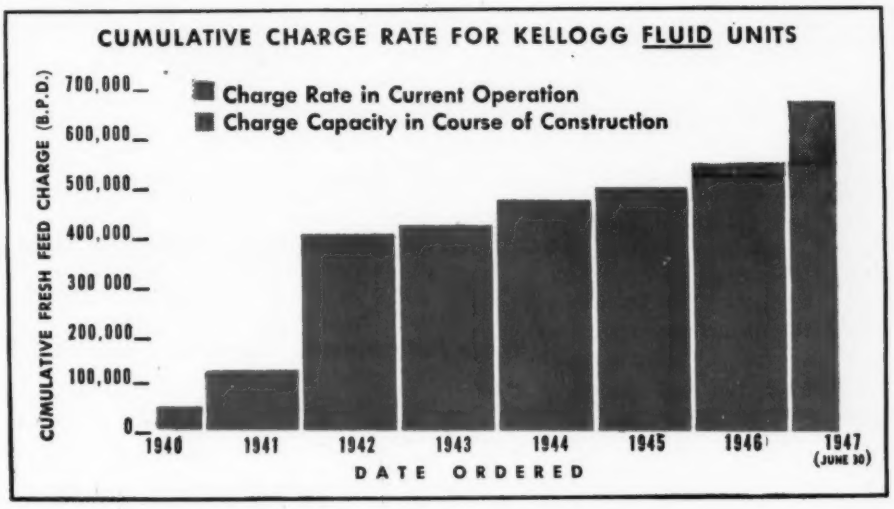
SINCE 1940 the petroleum industry's preference for Kellogg *Fluid* "Cat" Units has been growing by leaps and bounds — and for good, sound reason. *Fluid* "Cat" Crackers built by Kellogg deliver a combination of advantages available in no other catalytic cracking process.

1 EXTREME FLEXIBILITY...Kellogg *Fluid* "Cats" can handle the widest variety of feed stocks with equal efficiency, ranging from kerosene to the heaviest gas oils, and even to reduced crudes.

A flick of the wrist changes the catalyst to oil ratio, the temperature, the bed levels, or the degree of re-cycling—*enabling the refiner to vary product quantity and quality to meet changing market demand.*

2 GREAT ECONOMY OF OPERATION . . . because Kellogg *Fluid* "Cats" have demonstrated on-stream efficiencies as high as 95.3%. One large unit recently ran 428 consecutive days and was turned around in 21 days.

3 HIGHEST EARNING POWER is the direct result of the flexibility, efficiency and economy of the Kellogg *Fluid* "Cat" . . . adaptable to big or little refineries, alike. Kellogg *Fluid* "Cats" are operating in refineries where the total crude capacity runs less than 10,000 BPD — as well as in some of the world's largest plants.



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What's Happening!

NOVEMBER 1
1947

HIGHLIGHTS OF THE NEWS *Various programs are being proposed for voluntary co-operative effort of the industry to meet the shortages of products now being faced in the east . . . . Standard of New Jersey says it may have to allocate its residual fuel oil supplies this winter . . . . Nearly all alkylate production will have to go into av-gas to meet increased military requirements . . . . First test of validity of Fluid catalytic cracking patents comes in suit filed in Delaware Federal Court . . . . General Motors discusses with oil men in Detroit tentative plans to introduce car with 8-1 compression ratio engine in '48 or '49.*

New Plant Construction Stanolind Oil & Gas Co. will build cycling plant in Bee county, Texas, to handle 159,000,000 cu. ft. daily of high pressure gas and an additional 7,000,000 cu. ft. of casinghead gas from the recently unitized Burnell-North Pettus field. Liquid products will include propane, butane, motor fuel, kerosene and fuel oil. Jones & Laughlin Supply Co. is design and construction engineer . . . Warren Petroleum Corp., in partnership with Carter Oil, Cities Service Oil, J. E. Crosbie, Inc., and Sinclair Prairie, will build natural gasoline plant at Maysville, Okla., to ultimately process 185,000,000 cu. ft. of gas, recovering 14,000 b/d of liquid products. Cost is $8-$10,000,000.

Standard Oil Co. of California is to build new 22-story office building in San Francisco adjoining present headquarters. New structure, to cost $4,000,000, will be same height as present building and match it architecturally. Improvements to company's El Segundo refinery, including package shipping warehouse, barrel reconditioning plant, utility buildings, spur tracks and 8-in. wharf pipeline will cost $2,500,000.

Daily production of 35,000,000 cu. ft. of casinghead gas, formerly flared in the Tomoconnor oil field near Refugio, Texas, started flowing to market when Humble Oil & Refining started operating the compressor section of the $3,000,000 natural gasoline plant being built jointly by Humble and Quintana Gas Co. When finished late this year the plant will strip 60,000 gals. daily of natural gasoline, LPG and commercial propane.

J. S. Abercrombie & Magnolia Petroleum Co. are spending $1,500,000 to increase crude stabilization and separator capacity, and to revamp absorption system at their Old Ocean (Texas) natural gasoline plant. . . . Shell Oil Co., Inc., and Coltexo Corp. will enlarge their jointly owned gasoline plant in the Wasson field, West Texas, to process an additional 24,000,000 cu. ft. of gas daily. At present 75,000,000 cu. ft. is being processed. . . . Barnsdall Oil Co. to build natural gasoline plant at Newhall, Cal., to process 40,000,000 cu. ft. per day of gas into natural gasoline and LPG. Engineering and construction contract to Refinery Maintenance Co., Compton, Cal. . . . Continental Oil Co. is taking options on land near Corpus Christi in case of future expansion needs but has no immediate plans to build plant there.

New Plants Abroad New 20,000 b/d refinery to be built in Holland, near Rotterdam, by California-Texas Corp., joint subsidiary of Standard Oil Co. of California and The Texas Co. Equipment will include thermal cracking and reforming, to produce gasoline, kerosene, gas oil and fuel oils. Plant, to process Saudi Arabian crude, to cost $18,000,000. Arthur G. McKee & Co. engineering and construction contractors. . . . William Barnes & Co., Los Angeles engineering firm, has begun dismantling the former Canol refinery at Whitehorse, which will be moved to Edmonton and erected there for Imperial Oil Ltd., which purchased it. . . .

Engineering and purchasing in progress for 30,000 b/d refinery for Petroleos Mexicanos, at Salamanca, to be built by Arthur G. McKee & Co. Costing $12,000,000, plant will include crude distillation unit, thermal cracking plant, treating facilities, Ethyl blending unit, tankage, offices and warehouse. Poza Rica crude will be processed. . . . British American Oil Co., Ltd., will spend $15,000,000 to enlarge its Montreal refinery to step up production of high octane motor fuel and distillate fuel oils; $2,500,000 to expand lubricating oil plant at Clarkson, Ont.

General First challenge of the patents on Fluid catalytic cracking is in suit filed in Federal District Court in Delaware, in which Alamo Refining Co. (Phillips Petroleum subsidiary) asks declaratory judgment of non-infringement of Fluid patents covering operation of the Sweeney, Texas, refinery which it purchased from WAA in June. Defendants are Shell Development, Standard Oil Development, Texaco Development Corp., M. W. Kellogg Co., International Catalytic Oil Processes Co., and Universal Oil Products and M. W. Kellogg as agents of Standard of Indiana. . . . General Motors disclosed to a group of oil men in Detroit tentative plans to put on the market in late '48 or early '49 Cadillacs and one model Oldsmobile with an 8-1 compression ratio, requiring gasoline with a 93 Research octane number. Majority were of opinion this move would adversely affect over-all supply of petroleum products. . . .

Federal Power Commission has issued permanent certificates to Texas Eastern Transmission Corp. for operation of Big and Little Inch pipe lines as gas carriers, as means of relieving gas shortage in Appalachian area. Company has been operating the lines on temporary certificate since last May. With additional compressor stations now planned, capacity of lines will be increased to 340,000,000 cu. ft. daily by Feb. 1, '48, and to full capacity of 435,000,000 cu. ft. by Aug. 1. . . .

U. S. is insisting that the 16 European nations in the Marshall plan justify down to end-use their estimates of petroleum and equipment requirements through 1951. Estimates of petroleum requirements were double deliveries to these nations before the war. Feeling is present figure can be scaled down materially without endangering the program. . . .

Ethyl Corp.'s contracts with refiners for tetraethyl lead supplies in 1948 call for meeting competitors' prices and permit customers buying outside if Ethyl cannot meet the needs. Contract price is 49.482 c/lb., with

reservation to change on 60 days' notice. Company states it expects sufficient supplies in 1948 to deliver from 110 to 120% of '47 shipments to customers contracting their entire requirements. . . .

Stockholders of Pacific Western Oil Corp. and Mission Corp. will vote in December on proposal to merge their companies into Sunray Oil Co. Skelly Oil Co., 59% of whose stock is owned by Mission, will continue as separate entity if the merger goes through . . .

WAA has sold more than three-fourths of the government-owned, war-built aviation gasoline facilities, which cost $260,000,000, and expects to sell four remaining refineries and three toluene plants before Jan. 1. Of the total 33 plants built, 22 have been sold, two leased and two withdrawn from the surplus property list and turned over to Rubber Reserve Corp. The 22 plants sold brought $52,700,000.

Stretching Supplies Interior Secretary Krug has called on the National Petroleum Council to consider measures the industry can voluntarily take to conserve petroleum supplies or increase their availability. . . . Conference of Atlantic Coast marketers says it will ask Congress to consider nation-wide gasoline rationing, to provide greater transportation facilities for heating oil. . . . Program for scheduling flow of crude and products, for coordination of transportation facilities and for study of means to maximize distillate fuel production and to increase gasoline supplies by lowering octane numbers for motor gasoline is proposed by B. L. Majewski, vice president of Deep Rock Oil, and API vice president of marketing. . . . Socony-Vacuum official says company may have to ration gasoline and heating oils this winter. . . . Standard of New Jersey official says it may have to allocate residual fuel oil supplies at 10 to 15% less than in winter of '46-'47.

Military Requirements Latest estimates of U. S. military requirements for fiscal year ending June 30, '48, by NPC subcommittee, call for total of 115,500,000 bbls. of major products. This is 10% larger than estimate of last May. Requirement for "avgas" is 20,702,000 bbls., 33.6% higher than earlier estimate. Most of the increased demand will be for 115/145 grade. Nearly all the industry's planned alkylate production must be blended into "avgas" if the new requirements are met. Estimate of motor gasoline requirements for military is 13,690,000 bbls., 38.6% higher than earlier estimated figure. Requirements for other major product are placed at 81,107,000 bbls., about the same as the earlier figure.

Foreign First cargo of Middle East (Kuwait) crude arrived at Socony-Vacuum's Paulsboro refinery in temporary program to ease company's current short position on crude and enable it to continue peak runs at refineries. At least two other cargoes to follow, making 8500-mile trip via Suez Canal. . . . Deep Rock Oil Corp. has become one of the partners in the American Independent Oil Co., bringing to 12 the number of independent firms participating in this recently announced foreign petroleum venture, of which Ralph K. Davies is president.

Standard-Vacuum Petroleum Maatschappij refinery at Palembang, Sumatra, heavily damaged during war, has started limited operations and was to be back on stream running 20,000 b/d of crude by end of October. Full prewar operating rate will not be reached until late in 1948.

Research Cities Service Research and Development Corp. has been formed to centralize and expand research activities of individual Cities Service operating companies. New laboratory may be built in New York area. A. P. Frame will be president of new company. . . . ASTM Advisory Council approved as tentative standard the Research Method for determining knock rating of motor fuels and the Supercharge Method for knock characteristics of aviation gasoline, as proposed in the report of Committee D-2. . . .

An improved method for determining the efficiency of jet engines, by a new method for analyzing exhaust gases, has been developed at the jet engine research laboratory of Shell Oil Co., Inc., at Wood River. The new analytical techniques are available to other oil companies studying jet fuels.

New technical committee organized by the ASTM to work on problems pertaining to standardization of automotive anti-freezes; among them standardization of test methods for examination and evaluation, investigation of corrosive properties. H. R. Wolf, Research Laboratories Division, General Motors Corp., is chairman. . . . Seven new standard samples of hydrocarbons announced by National Bureau of Standards as available for test purposes bring to 119 the total of hydrocarbons available through NBS. The seven are 2, 2, 4-trimethylhexane; 2, 3, 3-trimethylhexane; 2, 3, 5-trimethylhexane; n-butylcyclopentane; 1-hexene; trans-2-hexene; 2-ethyl-1-butene.

Names in News John S. Wertz, credit manager and secretary-treasurer of Vickers Petroleum Corp., elected president to succeed C. L. Henderson, recently deceased. . . . Richard Funkhouser, petroleum geologist and until recently petroleum attache at U. S. Embassy at Paris, is to be U. S. petroleum observer in Middle East, with title of third assistant secretary of embassy at Cairo. . . .

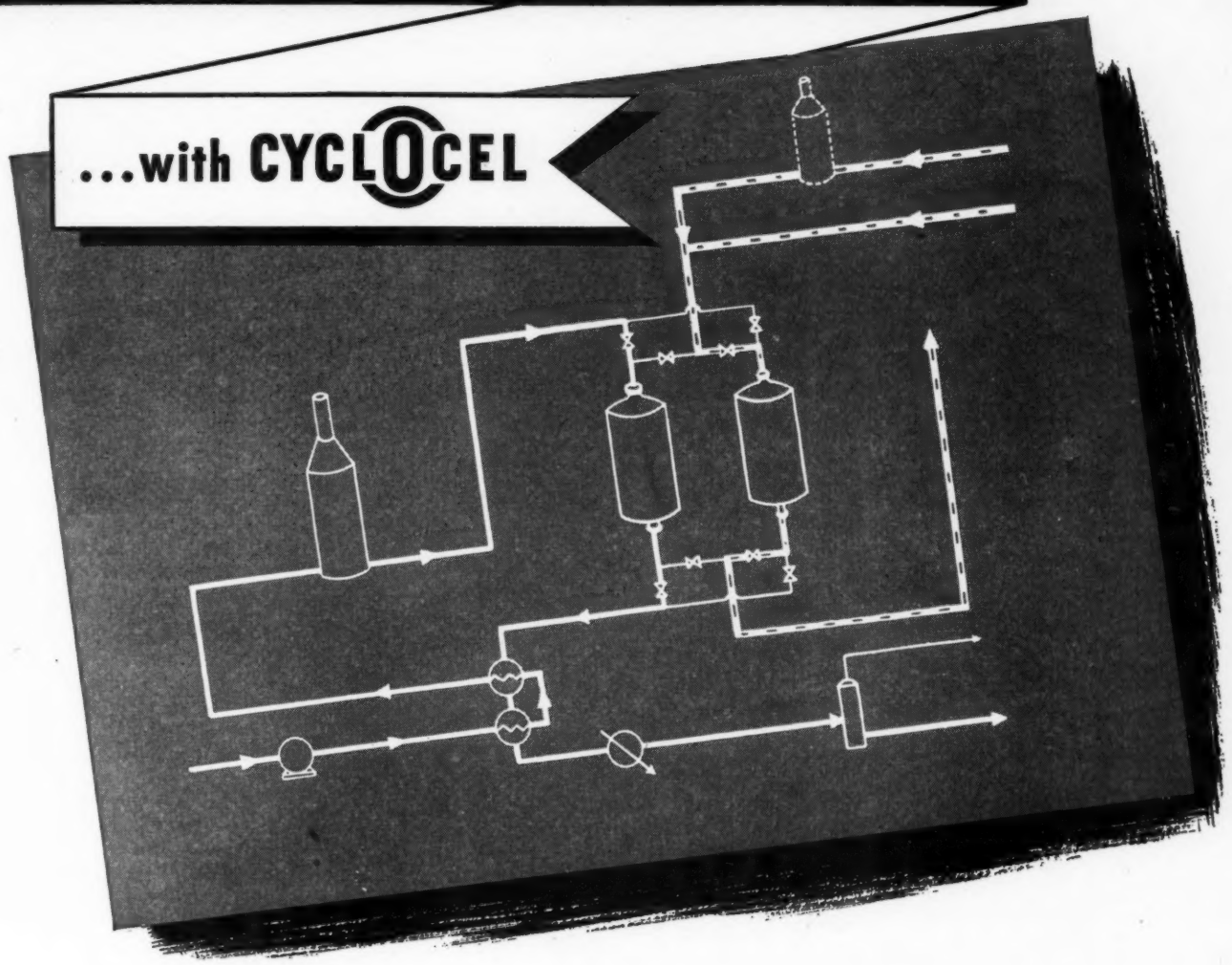
A. P. Frame, vice president of Petroleum Advisers, Inc., New York (Cities Service) is president of the new Cities Service Research and Development Corp. . . . K. H. Crandall, formerly president of The California Co. (Standard of California), has been made vice chairman of the board. H. C. Teasdel, former vice president, becomes president. . . .

Carl E. Cummings, head of The Texas Co.'s Beacon research and development laboratories, is chairman of a permanent API liaison committee with the Army. . . . J. P. Patterson, Pan American Petroleum & Transport, heads the reactivated NPC committee on tankers; Claude P. Parsons, Well Protection Inc., Houston, the committee on oil industry materials requirements. . . .

E. L. De Golyer, prominent petroleum geologist, Dallas, named member of an advisory committee for exploration and mining of Atomic Energy Commission. . . . Dr. Gustav Egloff, research director for Universal Oil Products Co., Paul Foote, executive vice president Gulf Research and Development Co. and Dr. Robert E. Wilson, board chairman, Standard of Indiana, appointed by the Atomic Energy Commission to a board of consultants to study problem of developing useful power from nuclear energy. . . .

More high octane gasoline at *Low Cost*

...with CYCLOCEL



Cyclocel—desulfurization and reforming catalyst—can be your *low-cost* answer to the improvement of clear and leaded octane numbers. Smaller refiners, in particular, can use Cyclocel for the upgrading of present light oil quality, with relatively little additional investment. In all cases, Cyclocel is valuable in reducing tetraethyl lead costs and in easing problems which arise from running higher-sulfur crudes.

Here are pertinent facts about Cyclocel:

LONG LIFE—Cyclocel's uniform granules are rugged. They retain their shape and catalytic efficiency under severe conditions of flow and temperature. In processes embodying reforming and desulfurization, catalyst life commonly reaches two years with negligible loss of activity. In non-regenerative desulfurization systems, throughputs up to 15,000 b/ton are realized.

SELECTIVITY—Cyclocel does not promote undesirable side reactions.

LOW-COST—Cyclocel's simplicity of use and exceptional stability make for low operating costs.

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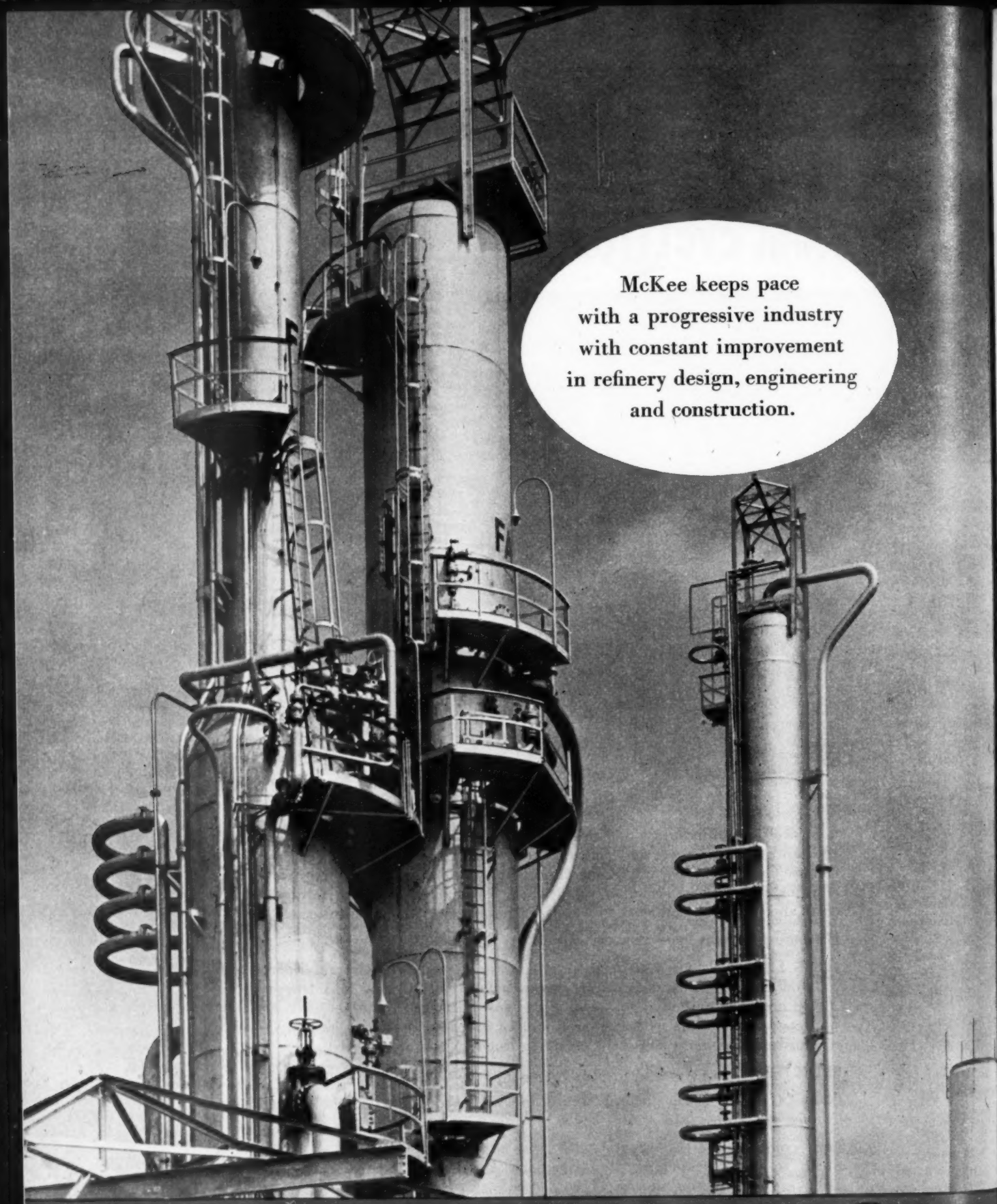

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TOMORROW

... IN PETROLEUM TECHNOLOGY

Current technological and economic developments discussed here in the light of their future bearing on petroleum refining operations include:

- **Petroleum import prospects not necessarily gloomy.**
- **Xylene booklet gives data on properties, uses.**
- **Technical data on Catarole process recently revealed.**
- **Another oil company markets new petroleum detergent.**
- **Government issues chemical statistics directory for 1945.**

Petroleum Import Prospects Not Necessarily Gloomy

AS THE petroleum industry well knows, this country in late 1946 departed from its historic role as a net exporter of petroleum. "A gradual decline in the productive capacity of the crude oil producing industry, concurrent with a continuous growth in actual and anticipated demand, has induced a general acceptance in many quarters of the hypothesis that to an ever-increasing extent we shall have to rely upon foreign sources for our petroleum needs . . . a public statement last year by certain Government officials that we will be importing half our requirements by 1965 has impressed itself on the public mind because of the apparent extremity of its view." (1)

All Government prognosticators, however, do not agree, as the reference just quoted proceeds to show. Space does not permit details of the argument presented, but it is proper to note that Trisko strongly objects to the following 1965 U S. forecast:

Production (1,000 bbl.):	
Crude oil	1,232,500
Nat. gasoline	100,000
Imports of crude and refined oils	1,232,500
Total supply	2,565,000
Exports of crude and refined oils	10,000

The gist of his argument is that foreign sources, while possessed of abundant reserves, are unlikely to produce any such excess as 1,232,500,000 barrels annually by 1965, especially as regards refined products; that lessened demands for residual fuel oil combined with higher efficiencies in machines and burners using petroleum products will reduce the above total requirements by 250,000,000 barrels and—more important—that petroleum substitutes will provide 620,000,000 barrels of oil annually by that time, a factor disregarded by the other forecaster.

Three of Trisko's tables are of particular interest, although their presentation without textual qualifications is unfortunately necessary.

(1) Trisko, R. L., *United States Petroleum Import Prospects*, U. S. Department of Commerce, Industrial Reference Service, Volume 5, Part 11, Number 2, 1947. 14 pages.

TABLE 1—Comparative Refinery Prices for Gasoline from Petroleum, Coal or Shale, and Natural Gas (Dollars per barrel)

Item	Range	Median
Refined gasoline, 73-75 octane, March 19, 1947*	$3.05–$3.57	$3.31
Estimated crude oil price†	1.70– 2.00	1.85
Gasoline from shale or coal, estimated selling price‡	3.78– 4.62	4.20
Estimated competitive crude oil price	2.11– 2.58	2.35
Gasoline from natural gas synthesis, estimated selling price‡	2.94– 3.57	3.26
Estimated competitive crude oil price	1.64– 2.00	1.82

* *National Petroleum News*, March 19, 1947.
† Author's estimate.
‡ Includes 63c per barrel for profit and return on investment.

TABLE 2—Estimated Landed Cost in United States of Foreign Crude Oil (Dollars per barrel)

Item	Arabian	Venezuelan
Cost of production*	$0.30	$0.50
Royalty and taxes	0.21	0.35†
Transportation‡	.75	.20
United States import tax	.105	.105
Difference in refinery realization	.20	.10
Profit	.25	.25
Total cost	$1.815	$1.505

* Maximum estimates; current costs probably approximate no more than 15c in Arabia and 35c in Venezuela.
† Estimated, as of 1946.
‡ Assuming company-operated tanker and completion of pipeline to Mediterranean.

TABLE 3—Suggested Supply and Requirements of Liquid Fuels in United States, 1965 (In million of barrels)

Estimated requirements	2,565	
Less contraction of residual fuel oil consumption and increased efficiencies in consumption of other products	250	
Probable actual demand	2,315	
		Percent
Suggested supply of liquid fuels*		
Domestic crude oil production	1,230	53.1
Imported crude oil and products	365	15.8
Natural gasoline	100	4.3
Synthesis of natural gas	175	7.6
Shale oil and/or synthesis of coal	445	19.2
Total	2,315	100.0

* Assuming a decline in crude oil output to the level indicated; to the extent that this decline is retarded, resort to other sources will be diminished.

Many technologists will disagree with Trisko's assumption that 445,000,000 barrels of "oil" will be produced from shale oil and/or synthesis from coal as early as 1965, but his bulletin serves as a reminder that all forecasters do not necessarily agree that this country will shortly be completely at the mercy of foreign sources for its vital supply of oil. The figures presented, however, despite this message, are adequate evidence that this situation can be prevented only by improving use efficiencies, developing processes and building plants to produce petroleum substitutes, and increasing domestic exploration efforts.

Xylene Booklet Gives Data On Properties, Uses

IN THIS age of burgeoning journals and library shelves, any good index to the information on an important subject is always welcome, no matter why it was prepared. Not that there is anything dishonest in advertising a product by disseminating information concerning it—only the recency of this technique invites attention to its undoubted value. Many companies are now promoting increased use of their products in this manner, and a recent booklet (2) on the xylenes — important new "chemicals from petroleum"—is an excellent example of this technique.

An "outline of the literature on the three isomeric xylenes," the booklet treats each isomer separately, summarizing chemical and physical properties with extensive reference to a bibliography of 410 items. In addition, each section contains a diagrammatic chart of possible uses

As mentioned recently in this feature and elsewhere, the production of xylenes from petroleum—by hydroforming or polymerization—is a development of considerable significance. This booklet provides the

(2) Anon., *Xylene Technical Review*, Oronite Chemical Company, San Francisco, 1947. 32 pages.

technical background for future uses which may add to the importance of these aromatic hydrocarbons.

Technical Data on Catarole Process Recently Revealed

THE petroleum and chemical industries in England and this country were highly interested, last year, in the announcement by Petrochemicals, Ltd., of their intention to build a large plant to produce aromatic hydrocarbons from petroleum by the "Catarole" process. A very wide range of aromatic products were listed, but little technical data were revealed.

It is therefore of interest to note that a recent paper[3] presented before the British Institute of Petroleum sheds more light on this subject and also constitutes an excellent review of the technical data on the production of "aromatics from petroleum."

As Steiner mentions, there are two fairly distinct methods of producing aromatics from petroleum: (1) hydroforming or catalytic reforming, "based on such chemical reactions as dehydrogenation, cyclization, and isomerization, and characterized by the fact that a given nonaromatic hydrocarbon is converted into the corresponding aromatic compound having the same number of carbon atoms," and (2) high temperature cracking, with or without catalysts, "based on a much more radical decomposition into olefins and diolefins of low molecular weight, from which, in a second step, aromatic compounds are built up by condensation and polymerization reactions.

"Since in this (latter) process cracking and condensation reactions are involved, there is no rigid correspondence between molecular sizes of charge and product. In general, a wide variety of charging stocks, from natural or cracked gases to high boiling gas-oils, can be used, and the products contain the full range of aromatic products from benzene to highly condensed rings."

While the "Catarole" process is not mentioned by name, it is described in some detail as falling in the second of the above categories. Conducted at 650-680° C., with "metallic dehydrogenation catalysts, . . . at low pressure, and at space velocities of about 0.3-0.4 vol./vol./hr.," the process is "carried out in cycles of 'on stream' periods of some 50-60 hours. . . . After a regeneration period of 10 hours, the plant is put 'on stream' again. . . Any charging stock boiling up to 300-350° C. can be used, and one is limited only by carbonization troubles, which increase with the boiling range of the feed. . . . In general, the yield of aromatics (liquids) increases with increasing boiling range of the charge," and the higher this boiling range, the greater the yield of alkylated aromatics and polycyclic aromatics.

Petroleum will certainly be used to an increasing extent as a raw material for the synthesis of aromatic hydrocarbons, and the "Catarole" process is an interesting example of specific methods for their production. Economic data on such processes will be welcomed with more than idle interest.

(3) Steiner, H., *Journal of Institute of Petroleum* 33, No. 283, 410-38 (1947), "Aromatics from Petroleum."

Another Oil Company Markets New Petroleum Detergent

AS has been mentioned frequently in this series, more and more petroleum companies are entering the field of detergents manufacture. George L. Parkhurst of the Oronite Chemical Company pointed out in his recent speech before the Manufacturing Chemists' Association that an "important aromatic by-product of petroleum refining are the sulfonates —complex mixtures of sulfonated hydrocarbons which have found many uses and which are in short supply.

"Due to the inherent limitation of such by-product operations, the petroleum industry, following the lead of the chemical industry, has turned to synthetic methods. Thus by-product sulfonates led to synthetic alkyl aryl sulfonates, useful as detergents."

These remarks were certainly not intended as an introduction to the announcement of a new detergent by another company, but they apparently fit it very well. According to a release last month, the Daugherty Refinery, a division of L. Sonneborn Sons, Inc., will produce a water-soluble petroleum sulfonate called "hydronate" for sale to industrial producers of detergents for their further processing into finished products.

Synthetic detergents are said to have three deficiencies: (1) they are not completely satisfactory for washing heavily-soiled cottons; "soap itself is not too satisfactory," however; (2) while they are superior to soap in hard waters, they do not in general hold an edge for use in soft waters; and (3) they are difficult to produce as "bar soaps," although competitive as regards powdered, granulated, or beaded compositions.

As Parkhurst points out, however, "even without complete solution to these problems the expansion (in volume of production) will be substantial." While "synthetic detergents will not . . . ever completely replace soap and will not equal soap in volume of sales within the predictable future, . . . population increase, and the increase in per capita use of washing compounds of all types, will probably be such that the gross amount of soap used will not show any long-term decline even if commercial success of the synthetic detergents is such as to fulfill not only our expectations but our fondest hopes."

Government Issues Chemical Statistics Directory for 1945

LITTLE need now be said about the value of market research to the manufacturers of chemicals. Because of statutory authority, many Government agencies are collectors of statistical data in this field, and their releases of such information are becoming so numerous that there is always a possibility that valuable ones will go unnoticed even when needed.

For this reason, a recent directory[4] of Government chemical statistics for 1945 should prove of considerable value, especially since it "is the first number of a new series in the field of chemical statistics. . . . Its purpose is to provide an index to the Government storehouse of chemical statistics" issued by the Department of Agriculture, Bureau of the Census, Department of Commerce, Department of the Interior (including the Bureau of Mines),

In his recent discussion[5] on the prospects for petroleum companies entering the chemical field, Bradley Dewey made the following remarks; it remains for petroleum companies to refute the truth of such statements by positive action, in which the proper use of statistics is vital:

"The petroleum industry will have to learn that market analyses, as applied to chemicals, are not what the oil companies have known by the same name. It is not a question of counting cards, predicting traffic loads, and evaluating brand preferences, service station locations, and the effectiveness of consumer advertising. In some cases it is nothing more than the hunch of a good organic chemist, but in most cases it has to do with the prediction of long-range trends and involves judging the markets for products to be made from the product that the company is going to sell—second generation sales.

"In all probability the chemical companies are better staffed to draw the right conclusions from the data presented by these surveys. For a long time to come they will have the best mixture of brains, willingness to think, imagination, and experience —experience gained from the manifold mistakes of the past."

(4) Anon., *Chemical Statistics Directory No. 1 (for 1945)*, Chemical and Drugs Section, Office of Domestic Commerce, U. S. Department of Commerce Industrial Series No. 66, Superintendent of Documents, Washington, 1947. 60 pages. 15 cents.

(5) Dewey, Bradley, *Chemical and Engineering News* 25, No. 34, 2340-41 (1947), "Is It Too Late"?

CONTROL...*Yes*

But

HOW EFFICIENT?

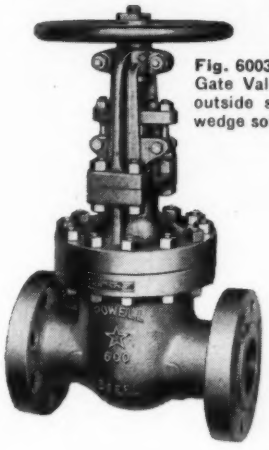

Fig. 6003—Class 600-pound Cast Steel Gate Valve with bolted flanged yoke, outside screw rising stem and taper wedge solid disc.

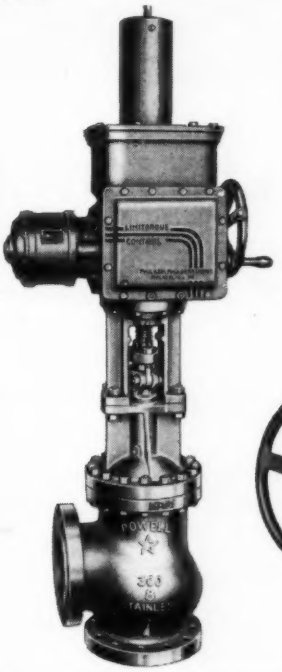

Fig. 3053 S. S.—Class 300-pound Cast Alloy Steel Angle Valve with top-mounted, explosion-proof, enclosed electric motor operator for quick, positive opening and closing.

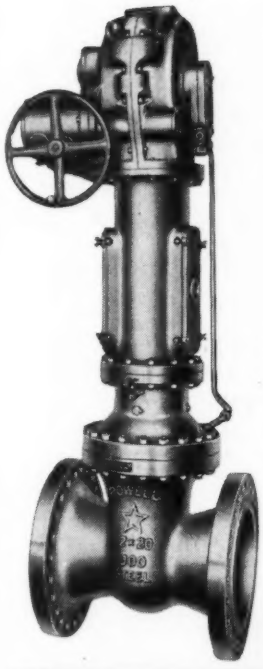

Fig. 3023 Mod.—Class 300-pound Cast Alloy Steel Gate Valve, designed and built to meet special requirements and to handle high temperatures. It has 12" port size venturied to 20" size end flanges to accommodate insulated pipe. Equipped with top-mounted, enclosed, explosion-proof electric motor operator for quick, positive opening and closing.

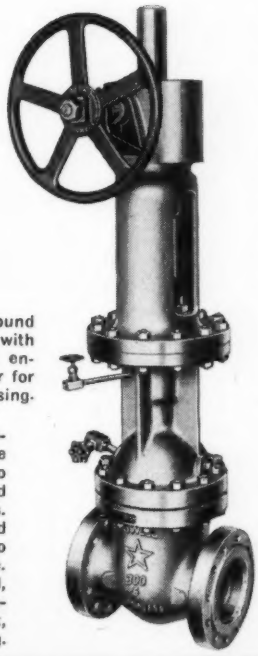

Fig. 3003 S. S. Mod.—Class 300-pound Cast Alloy Steel Gate Valve. Designed to withstand the ultra-high temperatures encountered in T. C. C. Has drain valves in stuffing box and bonnet and cooling fins to dissipate the heat, thereby reducing temperature in the stuffing box. Gear operated.

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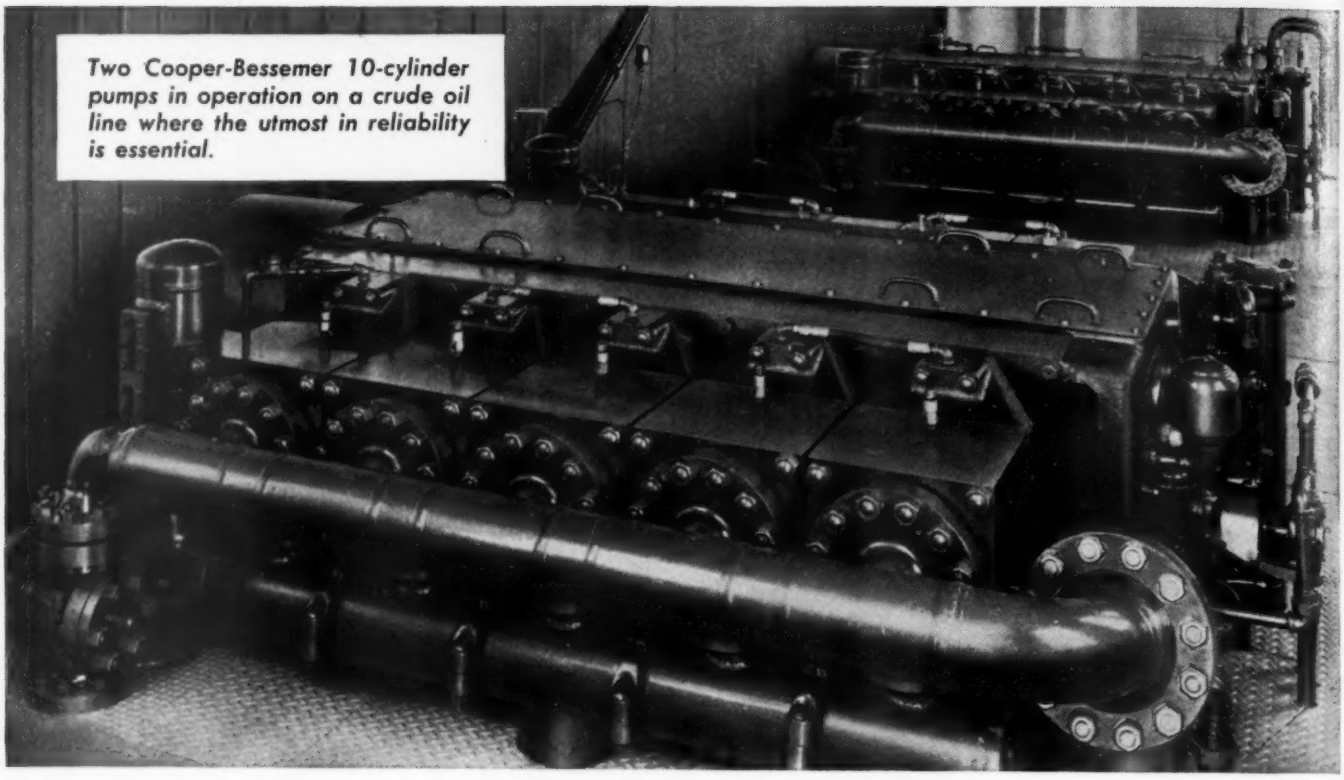

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PETROLEUM PROCESSING
NOVEMBER, 1947

Even if this bomb-blasted tankage at the Japanese Kudamatsu refinery had been intact, the Japs would have had nothing to put in it. A critical shortage of crude, among other things, contributed to their downfall

Desperate Japs Tried to Make 'Avgas' Even from Pine Tree Roots, Needles

By D. P. THORNTON, Jr. Assistant Editor

The Japanese navy carried on extensive research in fuels and lubricants not only from petroleum, but from substitute sources as well, it is brought out in a hitherto restricted report by a U.S. Naval Technical Mission, which went into Japan at the close of the war. In 1941 the Jap navy operated one of the largest petroleum research stations in the world, and two complete refineries. Their high octane blending agents for aviation gasoline were made by hydrogenation of cracked gasoline and the hydrocracking of gas oil.

When our subs cut off the supply of crude to the Jap home islands, their technical staffs tried to find means to make fuels from raw materials available there. One desperate effort was to retort oil from pine roots and to catalytically crack this oil and reform the product into aviation gasoline.

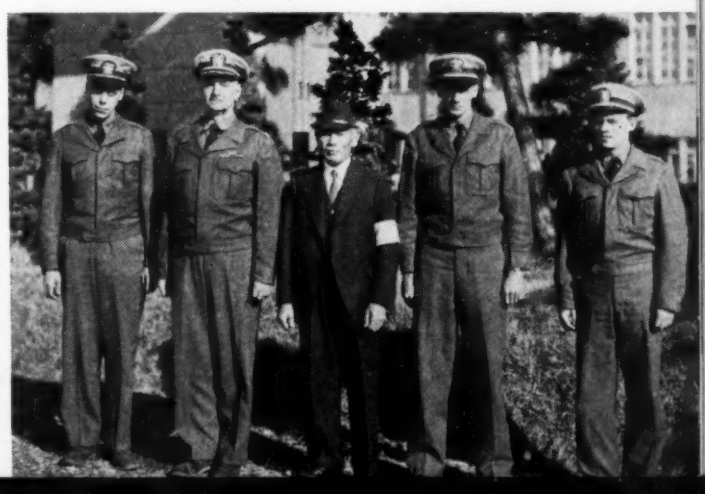
THE Japanese were not the grinning mimics they were popularly supposed to be when it came to fighting a war in which petroleum products were their life blood. Confronted with a truly desperate demand and unforeseen interferences with anticipated crude supplies, they demonstrated surprising ingenuity in developing petroleum substitutes. After the outbreak of the European war they received little technical help from Germany and still less from this country, which makes some of the petroleum developments found in Japan all the more remarkable.

The Japanese miscalculated grievously on many matters, but probably one of their most serious mistakes was underestimating the effect of the U. S. submarine blockade on their scheduled imports of petroleum from the East Indies. Their limited stockpile was quickly exhausted. Consequently, at the end of the war they were making desperate efforts to produce gasoline from natural rubber, alcohol, pine tree roots, coal, tar, pine needles and other unusual oil sources.

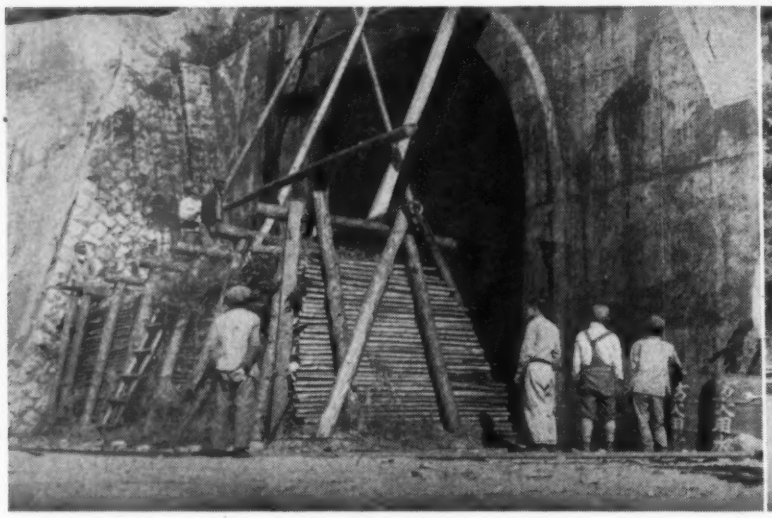
Their crude oil refineries were for the most part long since shut down for lack of charging stock; many ships and aircraft were perforce idle for lack of fuel; airplane pilots went into battle with inadequate training resulting from the shortage of gasoline; the only 100-octane gasoline they had was in laboratory quantities; bunker fuel was made from a blend of crude residuum and vegetable oils. The list could go on and on.

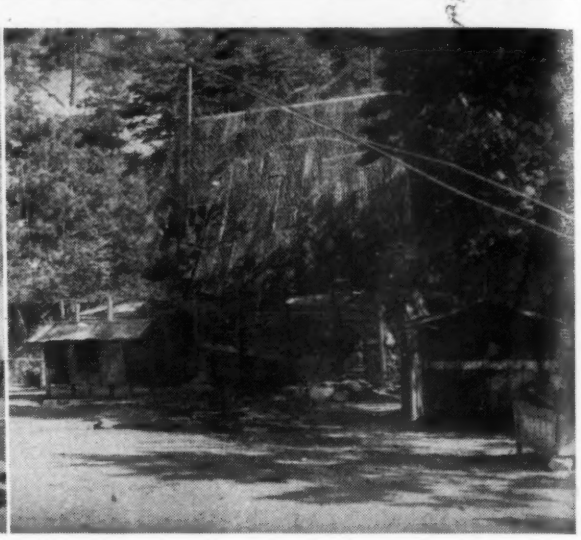
Much information on what the Japs were doing to meet their oil shortage is brought out in a hitherto restricted U. S. Navy report on the activities of the U. S. Naval Technical Mission to Japan (NavTechJap) entitled "Japanese Fuels and Lubricants". It was prepared by a four-man petroleum team consisting of Comdr. G. L. Neely, San Francisco, Standard Oil Co. of California; Lt.-Comdr. C. S. Goddin, Tulsa, Stanolind Oil & Gas Co.; Lt. W. H. Millett, Buffalo, Carbide and Carbon Chemicals Corp. and Ens. E. R. Dalbey, University of Washington, Seattle.

A significant feature of Japan's oil policy brought out in the report of NavTechJap is that the Japanese Imperial Navy did not depend on pri-



These are the men who investigated Japanese research and progress in petroleum. Left to right—Lt. W. H. Millett, Cmdr. G. L. Neely, Japanese Vice-Admiral N. Yamaguchi (last superintendent at Ofuna), Lt. Cmdr. C. S. Goddin and Ens. E. R. Dalbey





Underground storage for petroleum products was used extensively in Japan. These two views show the camouflaged entrances, with bomb blast protection walls at the Kure Naval Base

vate industry for either its oil research and development work or manufacture of refined products. On the contrary, the Navy, recognizing the critical position of the nation in this regard, was the most progressive petroleum group in Japan. It waged a constant battle trying to make the Empire self-sufficient in liquid fuel supplies.

The Japanese Navy first started research on fuels at Tokuyama in 1909, at the close of the Russo-Japanese war. By 1941 it had one of the world's largest fuels and lubricants research stations at Ofuna, Honshu, and operated two of the largest complete oil refineries in Japan. It also carried on a number of producing and refining operations at points outside the home islands.

The Japanese Army as well as private industry also carried on independent oil research.

Japan needed an estimated 30,000,000 bbls. of products annually for wartime military and civilian needs and in 1941 had refineries with an installed capacity of some 35,000,000 bbls. annually. Its own crude oil production was only about 2,500,000 bbls. per year. In the initial years of the war the Japanese planned to draw on their stockpiles of crude and refined products until such time as requirements for all needs could be met by development of a synthetic oil industry and imports of crude from the Netherlands East Indies. All refining was to be behind the "rings of defense" then being established.

At the time of the Pearl Harbor attack, the Japs had approximately 60,000,000 bbls. of crude and refined products in storage. Much of this was stored in large reinforced concrete tanks buried in the hills and completely camouflaged with the aid of heavy vegetation. A single large reservoir at the Sasebo Naval Base, for example, had a capacity of about 500,000 bbls. Total bulk storage at Tokuyama was about 9,000,000 bbls., with 4,000,000 bbls. at Sasebo and 1,000,000 bbls. at Kure.

What actually happened soon after the war with Japan started was that, even though production by mid-1943 from Borneo and Sumatra equalled expectations, U. S. submarines were becoming increasingly efficient in sinking Japanese tankers. The sub toll became so great that by 1944 Japan found her oil stocks rapidly declining and the time schedule for

TABLE 1—Principal Sources of Fuels and Lubricants for the Japanese Navy During World War II

Year	Aviation Gasoline	Motor Gasoline	Kerosene	Aviation Lubricants	Diesel Fuel	Bunker Fuel
1940	1)California Crude 2)Imported Iso-Octane	Calif. Crude	Calif. Crude	Imported Finished Lubricants	Kettleman Hills Distillate	1)Calif. Topped Crude 2)Oha (Sakhalin) Crude 3)Shale Oil
1941	1)California Crude 2)Imported Iso-Octane	Calif. Crude	Calif. Crude	Imported Finished Lubricants	Kettleman Hills Distillate	1)Calif. Topped Crude 2)Oha (Sakhalin) Crude 3)Shale Oil
1942	1)California Crude 2)Imported Iso-Octane	Calif. Crude	Calif. Crude	Imported Finished Lubricants	70% Tarakan (Borneo) Crude 30% Shale Oil	1)Sumatra and Borneo Cracked Residues 2)Shale Oil
1943	1)Borneo and Sumatra Crudes 2)Iso-Octane	Sumatra Crude	Borneo and Sumatra Crudes	Osage and Rhodessa Crudes	1)10% Tarakan (Borneo) Crude—30% Shale Oil 2)90% Tarakan (Borneo) 10% Fischer-Tropsch Liquid	1)Sumatra and Borneo Cracked Residues 2)Shale Oil
1944	1)Borneo and Sumatra Crudes 2)Iso-Octane 3)Alcohol (Training Planes)	1)Sumatra Crude 2)Ethyl and Methyl Alcohol	Borneo and Sumatra Crudes	Osage and Rhodessa Crudes	1)10% Tarakan (Borneo) crude—30% Shale Oil 2)90% Tarakan (Borneo) 10% Fischer-Tropsch Liquid	1)Sumatra and Borneo Cracked Residues 2)Shale Oil
1945	1)Borneo and Sumatra Crudes 2)Alcohol (Training Planes) 3)Iso-Octane 4)Pine Root Oil (Not used in service) 5)Investigation of Miscellaneous Substitutes as Fuel Sources. (Rubber, Vegetable Oil, etc.)	Ethyl and Methyl Alcohol	Borneo and Sumatra Crudes	1)Osage and Rhodessa Crudes 2)Investigation of Synthetic Oils from Other Sources. (Paraffin Wax, Shale Oil, Vegetable Oil, Rubber etc.)	1)10% Tarakan (Borneo) Crude—30% Shale Oil 2)90% Tarakan (Borneo) 10% Fischer-Tropsch Liquid	1)Sumatra and Borneo Cracked Residues 2)Shale Oil 3)Substitute Fuels, such as Soya Bean Oil 4)Investigation of Pine Root Oil

TABLE 2—Japanese Navy Petroleum Activities, Aug. 15, 1945

Naval Fuel Depot	Location	Activity
First	Ofuna, Honshu	Fuel and "Lube" Research
Second	Yokkaichi, Honshu	Petroleum Refining
Third	Tokuyama, Honshu	Petroleum Refining
Fourth	Shinbara, Kyushu	Coal Mining and Carbonization
Fifth	Heijo, Korea	Coal Mining
Sixth	Takao, Formosa	Petroleum Refining
	Niitaka, Formosa	Manufacture of Synthetic Lubricants (not completed)
	Shinchiku, Formosa	Iso-octane Synthesis, Butanol Fermentation (not completed)
101st	Samarinda, Borneo	Crude Production
102nd	Balikpapan, Borneo	Petroleum Refining

research and development greatly foreshortened. A program of building wooden tankers then was started. This was seriously affected by a never-relieved shortage of engines for propulsion and the project was abandoned.

By early 1945 all refineries depending upon imported crude had ceased continuous production. The Navy plant at Tokuyama, for example, obtained its charging stock by transporting Netherlands East Indies stocks from Singapore. The tanker "fleet" consisted of several aircraft carriers and one battleship. How desperate was the situation can be surmised from the fact that a battleship sunk in the battle of Okinawa had edible soya bean oil in its bunker tanks.

Table 1 lists the principal sources of fuels and lubricants for the Japanese Navy, 1940 to 1945. The last of the war was of special significance, since the Japanese had to seek fuels and lubricants from such non-petroleum sources as pine roots, alcohol (from sweet potatoes), vegetable oils, shale oil and coal.

Production of synthetic oil from coal was disappointing. It reached a maximum rate of but 114,000 tons a year which was far below that called for under the Jap's "Seven-Year Synthetic Oil Plan." The shale oil works at Fushun, Manchuria, produced more oil than the combined production from all coal conversion plants in the Empire.

Refining Facilities

The Japanese Navy first started refining petroleum in 1920, with the installation of the first pipe-still in Japan, at Tokuyama. This plant was gradually enlarged to a crude capacity of 9500 b/d. In 1940 construction was started on the Navy's 17,000 b/d plant at Yokkaichi. In 1941 the various fuel activities of the Navy were designated as naval fuel depots, and those in existence in 1945, with their function, are listed in Table 2.

The Yokkaichi and Tokuyama plants were modern installations, and together accounted for about 25% of the nation's total refining capacity. Both plants were complete, equipped to carry on crude distillation, thermal cracking, and hydrocracking. They also produced their own synthetic methanol, iso-octane, tetraethyl lead and made lubricating oils and greases. In addition, Yokkaichi had two small fixed-bed catalytic cracking units. The processes were largely American, although some were based on Japanese research. Equipment was principally built in Japan and outwardly resembled that of American manufacture. A "Cross" cracking unit at one refinery is shown in the photograph below.

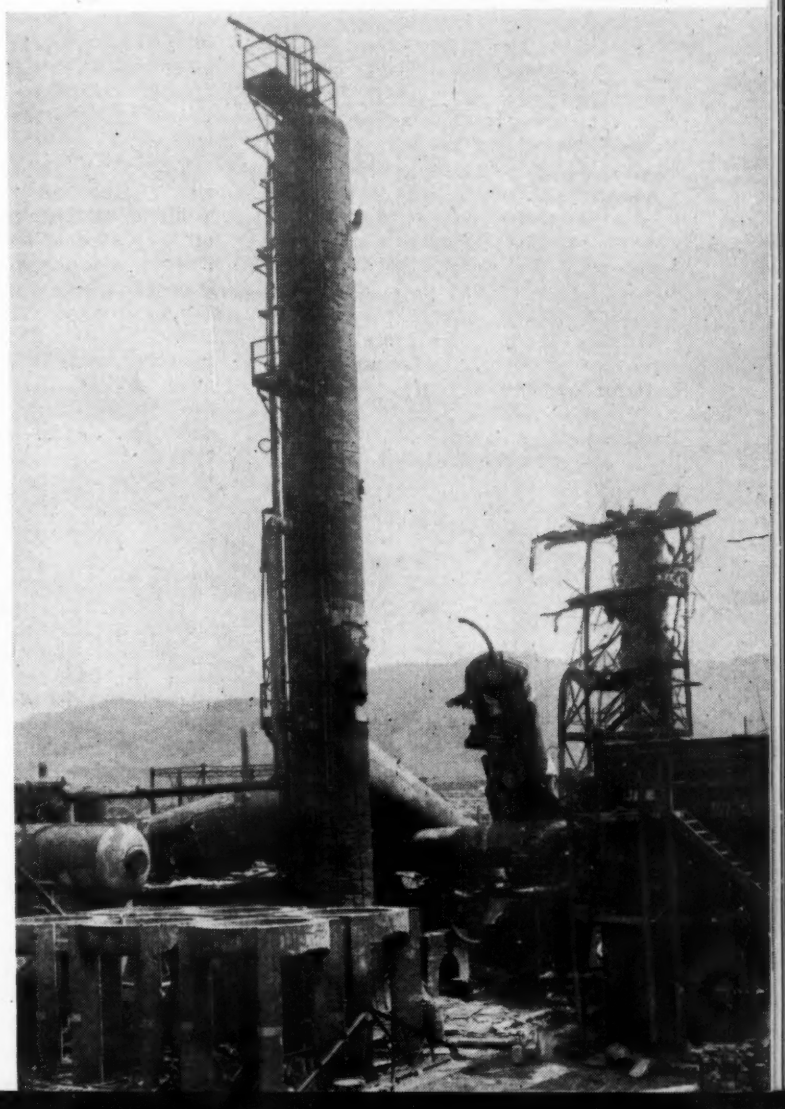
The facilities in Formosa and Borneo were etablished during the war. Part of the butanol fermentation facilities in Formosa were completed, but not those wherein butanol was to be catalytically converted into iso-octane. Likewise facilities there for synthetic lubricants manufacture still were under construction in August, 1945.

Aviation Gasoline

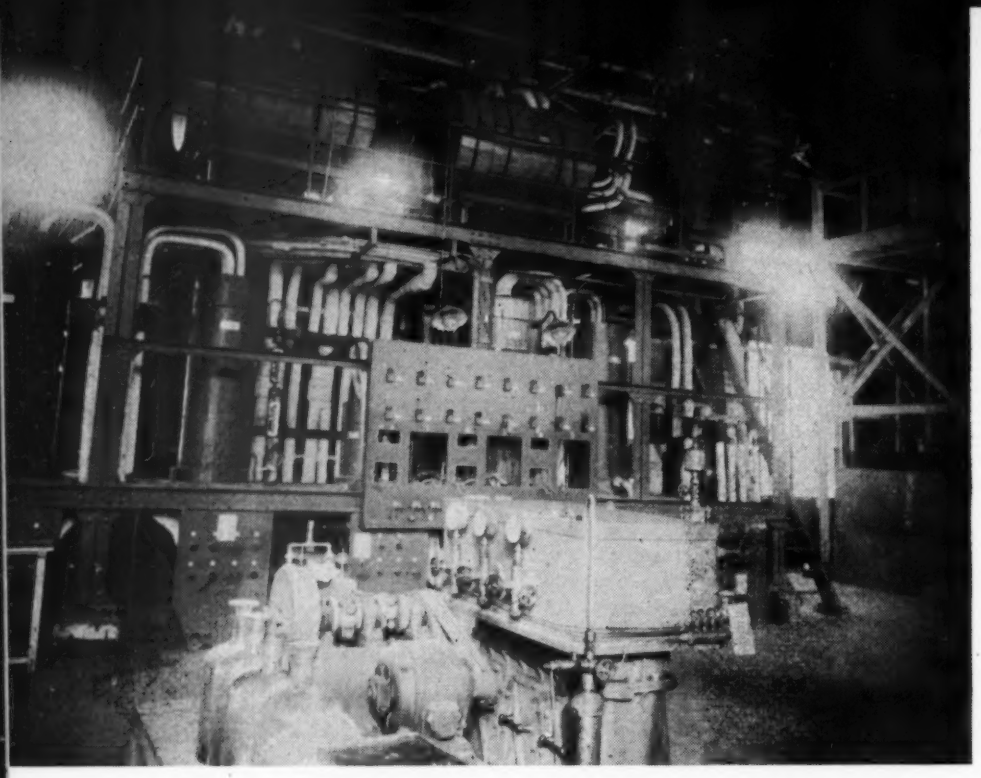
The air arm of the Japanese Navy used a 92-octane CFR Motor method combat aviation gasoline until 1942, when the rating was reduced to 91. This was maintained until the last few months of the war, when the rating was dropped to 87 for summertime use. Other wartime revisions included increases in the permissible maximum lead content and distillation temperature. One-hundred octane aviation gasoline was produced in experimental quantities only and never appeared in combat.

Iso-octane production in Japan at no time exceeded 80,000 bbls. annually, according to U. S. Navy estimates. This was due to relatively small supplies of the necessary C_4 hydrocarbons resulting from the limited cracking capacity of her tiny refineries. Most of it was synthesized from acetylene, in turn derived from calcium carbide, and from butanol produced by fermentation at plants located at Konan, Korea, and Yokkaichi, Honshu.

It was definitely established by the NavTechJap mission that our Moral Embargo in 1939 successfully prevented the Japanese from obtaining or purchasing American "know-how" on catalytic cracking. No evidence of Japanese application of the moving bed catalyst principle was ever discovered, for example. A fixed-bed cracking process was developed late in the war, using a domestic acid clay catalyst, but the actual production of aviation gasoline from this



Remains of a Japanese-built "Cross" cracking unit at Kudamatsu refinery, with foundations for never-installed Gray towers at left. The Japs used processes largely American in origin, with home-built equipment outwardly resembling that of American manufacture



Modern solvent refining processes were used by the Japs for manufacturing lubes, and they conducted extensive research studies on the subject. Shown above is a solvent extraction lube plant for pilot investigations at Ofuna

source was negligible. Their most successful processes for manufacturing high octane blending agents from the standpoint of the volume actually produced were the hydrogenation of cracked gasoline and hydrocracking of gas oils. These processes, as applied in Japan, were developed in the Naval Research laboratories.

The severe shortage of petroleum in 1945 stimulated intensive research on producing aviation gasoline from a variety of substitutes. These included pine root oil, soya bean oil, rubber, alcohol (from sweet potatoes), high and low-temperature coal tar, camphor, orange peel, birch bark and pine needles.

It was planned to install 36,000 pine root oil retorts in Japan with an anticipated annual output of some 2,500,000 bbls. of synthetic crude oil. By processing fractions of this oil in small catalytic cracking and reforming units, an annual output of 400,000 bbls. of aviation gasoline (90-92 octane with 0.15 vol-% lead) was expected. This program was well underway by the end of the war although the material reportedly never was used in combat.

The use of ethyl alcohol as a blending agent in aviation gasoline was impeded by the insolubility characteristics of 95% alcohol. Equipment for manufacturing anhydrous alcohol was very limited, effectively preventing any timely solution of the compatibility problem.

Diesel and Heavy Fuels

The NavTechJap Mission reports show the Japanese were fully acquainted with the advantages of high cetane Diesel fuels. However, none of their military specifications called for more than a 45-cetane rating. This requirement was reduced in 1942 to a minimum of 38, along with relaxed maxima on Conradson Carbon, ash, sediment and other particulars. In addition, they conducted research on 80 to 100-cetane number stocks, intending eventually to use them in special aviation and torpedo-boat Diesel engines which never materialized.

The Japs' chief source of submarine Diesel fuel during the war was a blend of Tarakan (Borneo) crude oil with 30% of refined shale oil from Fushun, Manchuria. Fischer-Tropsch Diesel oil, most of which was obtained from the Miike Synthetic Oil Plant at Omuta, was utilized by blending with 90% of Tarakan oil to meet specifications. About 50% of the Fischer-Tropsch Diesel oil produced in Japan was used in Army tanks.

Bunker oils were principally residual stocks from imported crudes and specifications were not unusual; before the war they came from California and afterwards from Borneo and Sumatra. Shale oils, while used for Navy bunker fuel, were never indiscriminately mixed or blended because it was found that paraffinic oils mixed with shale oils would precipitate in storage. Some oils of vegetable origin also were used in the closing days of the war.

Lubricants

The Japanese were on famine rations of lubricants, especially for aircraft engines, virtually from the start of the war. Planes grounded for lack of fuel actually relieved the lubricant crisis. Their native crudes were highly naphthenic, hence aircraft engine oils were produced from them only in small yields and most reliance was placed on imported crudes or finished oils. However, the Japs conducted extensive research with special emphasis on fundamental studies of the relationship between chemical structure and lubricant characteristics. This work was found to parallel that of several API projects in this country.

Their refineries used most of the modern U. S. methods in preparation of lubricants. Among the processes noted were propane deasphalting and continuous clay slurry contacting, such solvent refining processes as Duo-Sol and furfural, and dewaxing with acetone-benzene and benzene-ethylene dichloride (Bari-Sol).

One of the Japs' synthetic processes, used commercially, made aircraft oil by polymerizing a cracked paraffin wax distillate in the presence of anhydrous aluminum chloride. Likewise, considerable experimental work through the pilot plant stage was undertaken for the preparation of high-grade lubricants through polymerization of unsaturated fractions of shale oil. Fischer-Tropsch oils, vegetable oils, rubber and pine-root oils also were considered in this program.

One method, developed in the laboratory only, made a high Viscosity Index lubricant with good stability characteristics through the copolymerization of cracked wax distillate, naphthalene and sulfur. Radically differing from the practice in this country, although extensive research was conducted on pour point depressants, antioxidants, corrosion inhibitors and oiliness agents, additives were not used commonly in Japan.

Synthesis of Fuels from Coal

The Japanese had high hopes of producing much of their petroleum requirements by synthesis from coal, but their actual results were exceedingly disappointing. They initiated a Russian-like "Seven Year Plan" to achieve an annual production of 1,000,000 kiloliters (about 6,300,000 bbls.) each of synthetic gasoline and fuel oil by 1943.

Their peak year was 1944, but production then of all synthetic fuels actually amounted to only 114,000 kl., divided as follows: hydrogenation, 700 kl., Fischer-Tropsch, 17,800 kl., low-temperature carbonization 95,400 kl. The reasons for the dismal failure of their coal synthesis program were three: Lack of priorities for highly critical materials which were diverted to armaments construction; stoppage of equipment shipments from the U. S. and Germany resulting from our Moral Embargo and the commencement of war in Europe; operating difficulties best described as "process bugs".

Coal hydrogenation itself probably was studied more intensively in

Japan than in any country in the world except Germany. The Navy, with its usual policy of independence, flatly blocked purchase of patents covering Germany's Bergius hydrogenation process; instead it attempted to encourage "independence of thought" among its technical personnel by developing home-grown ideas.

With the maturing of war plans, they attempted to rapidly develop coal hydrogenation techniques on a commercial scale, in spite of the fact that the Navy process had not been thoroughly pilot-planted. The result was that only two commercial scale plants were built, neither of which achieved successful continuous operation. During the course of the war, the Japanese Army, discouraged with progress at home, attempted unsuccessfully to secure technical information on the Bergius process from Nazi Germany.

It is noteworthy that low-temperature carbonization of coal produced more synthetic liquid fuels than all other synthetic processes combined. Ironically, all successful coal carbonization installations were based on foreign designs, especially the Lurgi and Koppers systems. Japanese designed systems were installed, too, but were unsatisfactory and eventually abandoned.

Fischer-Tropsch synthesis was next largest in volume production but again their most successful plant, the Miike Works, was built directly on the basis of Ruhr-Chemie patents and all important equipment was of German manufacture. Japan's main contribution to the Fischer-Tropsch synthesis are Prof. Kita's synthetic iron catalysts, used in a section of the Takikawa plant throughout the war. (An iron catalyst is to be used in the two commercial Fischer-Tropsch plants now under construction or in design stages in this country.)

Japanese Research

One of the most impressive things learned about the Japanese, the NavTechJap reports states, is the tremendous importance they attached to research. In a land severely limited in resources, lavish expenditures of material and personnel for research were made down to the very last day of the war. The report continues:

"While in peace-time, American, German and British techniques were copied extensively both in laboratories and in refining installations, after the outbreak of the war the Japanese showed initiative in modifying known mehods to suit the changing conditons of raw material supply to produce the final products most needed.

"It has been concluded by some investigators that the research and scientific ability of the Japanese is mediocre. This is believed to be a fallacy. It is rather concluded, on the basis of research discussed herein, that their progress has been conspicuous and that within the next few decades Japan may well become one of the foremost technical nations of the world."

The Japanese Navy's First Naval Fuel Depot, located at Ofuna, was devoted exclusively to research, process development and practical testing of fuels and lubricants, and is an impressive demonstration of the Japanese emphasis on research. Prior to 1941, the Navy's research work was done at Tokuyama and several other depots. With the advent of World War II, the work was centralized at the Ofuna station, which continued in operation until August 15, 1945.

This station was one of the largest petroleum research centers in the world, covering approximately 100 acres and containing some 74 buildings, of which 37 were laboratories and the rest offices, shops and related facilities. Nearly all the laboratories were solid structures of steel and concrete, providing more than nine acres of working space. All wooden buildings were razed early in 1945 to minimize the fire hazard. Although none of these facilities ever suffered a bombing attack, elaborate precautions had been taken to protect key points.

Extensive underground shelters had been built and it was planned to move all of the "most critical" laboratories into these shelters. Photographs of the entrance to one of the underground laboratories and an interior view are shown on the next page.

It was admitted by station personnel that the large scale bombing attacks on surrounding cities and the constant air raid alarms severely interfered with the efficiency of Ofuna operations. It finally even became necessary to set aside a building for the manufacture of laboratory glassware since all commercial sources had been destroyed.

Ofuna was efficiently organized into four departments; Research, Experimental, Chemical Engineering

Part of the Ofuna Research Station at the Japanese Navy's First Naval Fuel Depot, where research, development and testing was centralized. Laboratories are in the middle area, with pilot plants scattered throughout. Underground labs were constructed in the hills in the background

and General Affairs (management, accounts, medical treatment and the like). Research was responsible for both research studies and pilot plant work; Chemical Engineering helped in the transition from laboratory-to-pilot plant-to-commercial operation and the Experimental Department was concerned with full-scale engine tests of fuels and lubricants.

Operations at Ofuna grew rapidly from the time of establishment in 1941 to the end of the war. In 1941 there was a staff of 45 men and a worker-complement of 1000; in 1943 there were 120 staff personnel and 1940 workers; in 1945, 410 staff men and 2800 workers. Most of the key personnel and department heads held Naval engineering specialist commissions and the depot at all times was commanded by rear- or vice-admirals. The NavTechJap report comments: "The personnel directing this research were competent chemists and chemical engineers, although there appeared to be a scarcity of skilled assistants and technicians."

About the first of August, 1945, it became apparent that the time would not be long before the Home Islands were invaded. On that date the extensive technical files at Ofuna were burned by order of the director, then Vice-admiral N. Yamaguchi. This made it necessary for NavTechJap, in the course of its investigation, to round up 100 of the key technical personnel and return them to the depot. There they were required to reproduce from notebooks, personal files, memory and other sources reports in English covering research activities during the war period. As these reports were prepared, they were reviewed with the authors by the petroleum team in the presence of interpreters and other specially skilled personnel. From this and other sources the NavTechJap reports were prepared.

Among the projects on which war time research was concentrated, the following are listed as evidence of Japanese creative scientific ability. These items are also thought to be of possible value to those conducting investigations along similar lines in this country.

1. Commercial application of a process for manufacture of iso-octane from acetylene.

2. Commercial application of a process by which terpenes are catalytically converted to aromatics.

3. Research on development of iron catalysts for Fischer-Tropsch synthesis.

4. Fundamental studies relating chemical structure to lubricating oil characteristics.

5. Commercial production of lubricants by polymerization of cracked wax distillates and research on co-polymerization of cracked wax distillates and research on co-polymerization of these distillates with various other materials.

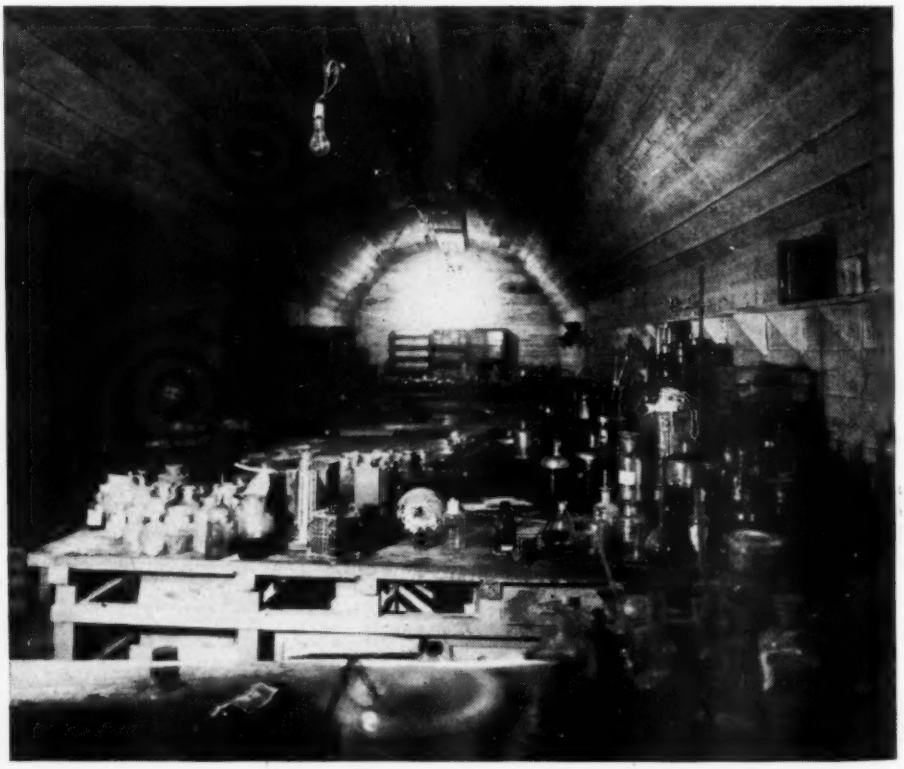
6. Commercial production of concentrated (80-85%) hydrogen peroxide using tin-lined and porcelain equipment.

The NavTechJap Reports

Report No.	Title*	Index No.
1	Fuel and Lubricant Technology	X-38(N)-1
2	Naval Research on Aviation Gasoline	X-38(N)-2
3	Naval Research on Alcohol Fuel	X-38(N)-3
4	Pine Root Oil Program	X-38(N)-4
5	Research on Rocket Fuels of the Hydrogen Peroxide-Hydrazine Type	X-38(N)-5
6	Research on Diesel and Boiler Fuel at the First Naval Fuel Depot, Ofuna	X-38(N)-6
7	Progress in the Synthesis of Liquid Fuels from Coal	X-38(N)-7
8	Naval Research on Lubricants	X-38(N)-8
9	Fundamental Hydrocarbon Research	X-38(N)-9
10	Miscellaneous Oil Technology and Refining Installations	X-38(N)-10

*All titles prefaced by "Japanese Fuels and Lubricants". Copies are available upon request from the Navy Department, Washington.

Entrance and interior of underground laboratories at Ofuna. Extensive shelters of this type were under construction or already built when the war ended



Instruments in the Modern Refinery

Installation and Maintenance Practices

By GEORGE A. LARSEN, Instrument Foreman
The Texas Co., Lockport, Ill.

Standardization of installation methods and arrangements, stocking a sufficient supply of standard repair parts, and provision of adequate shop facilities have resulted in an increase in departmental efficiency and appreciable savings in time and material in instrument maintenance at The Texas Co.'s Lockport (Ill.) refinery.

Included in the discussion are descriptions of a number of shop practices and shortcuts which have contributed much toward improved instrument services. This article is a condensation of a paper entitled "Instrument Installation and Maintenance Methods and Practices in a Modern Oil Refinery," presented by Mr. Larsen at the Second National Instrument Conference and Exhibit, Chicago, Sept. 10.

THE AUTOMATIC control of refinery processes becomes increasingly important as new and more complex methods are developed to meet the increasing demand for greater output of quality petroleum products.

These new processes have presented many problems to the control engineer, but instrument manufacturers have cooperated in developing equipment to meet the needs of the industry. Reliable instrumentation is so vital to the successful operation of our larger and more modern units that it is generally recognized that units of this type would be nonexistent without suitable controls.

Instruments are seen in general plant service today that only a few years ago were considered suitable for laboratory use exclusively. This increased tendency toward improved instrumentation is not a desire to "go modern" but is due to fundamental changes in equipment design, and a need for closer control of process variables for economic reasons.

Since instruments are now a "must" item in the modern refinery, there is a definite need for an independent, instrument department whose function is to specify, install, and properly maintain the plant instrumentation. It is the direct responsibility of this group to insure accurate and economical operation of indicating, measuring and regulating equipment.

In an effort to improve departmental and equipment efficiency at the Lockport refinery, certain standard practices and procedures have been adopted which have proved beneficial there and which may be of interest to other users or prospective users of instruments. It is not possible to discuss all these practices, so only those of major importance are being presented.

Shop Facilities

One of the most important requirements of an efficient instrument department is a well-equipped shop, centrally located with respect to the operating units. Good illumination for night or day work is very important where refinery operation is on a 24-hour schedule.

Fig. 1 is an interior view of the instrument repair shop, showing the excellent natural lighting for day operations. Fluorescent lighting over the large work table provides sufficient illumination for the night shift.

The work table is covered with 1/8 in. steel plate to permit the better handling of heavy equipment such as valves and meters. Lockers in this table and in the work bench along the walls provide each mechanic with

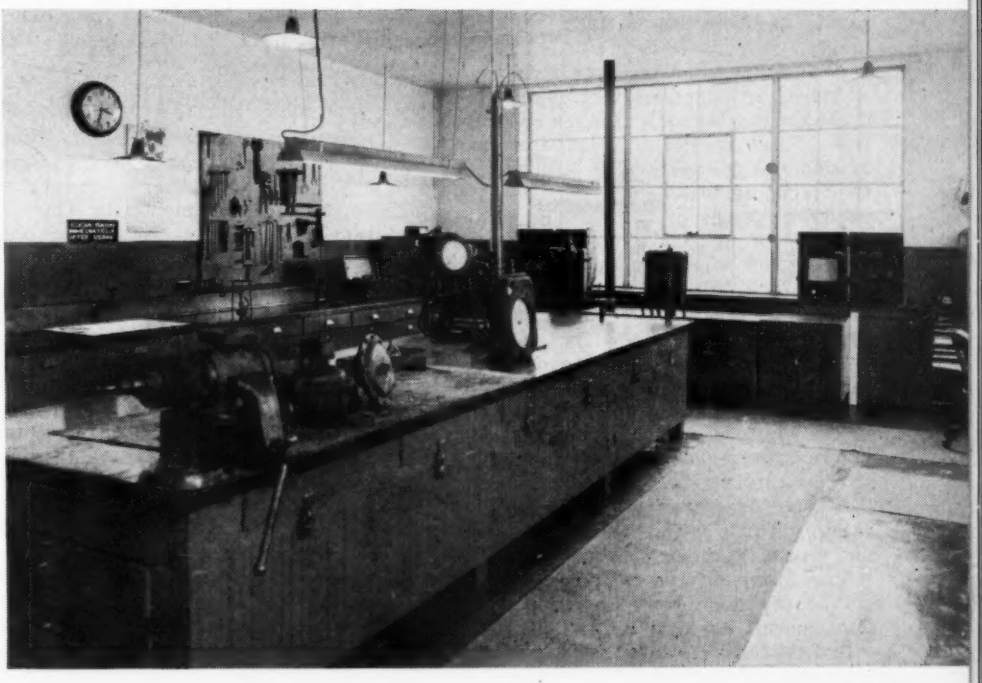


Fig. 1—Good lighting for both day and night work is an essential requirement of the well-equipped instrument repair shop. At The Texas Co.'s Lockport refinery natural daylight, incandescent ceiling drop lights and fluorescent lamps are all utilized

space for his small tools and personal effects.

One end of the central work table is equipped with two meter test racks and the necessary manometers, air connections, regulating valves, and flexible plastic tubing. Both racks are provided with drip pans to keep the table top and floor free of mercury.

Safety is a matter of primary importance to the instrument personnel. They are instructed in the safe handling of mercury, inflammable liquids, and the servicing of hot oil, light hydrocarbon, caustic, and acid meters. Metal safety hats are worn when working in areas where falling objects may cause an injury. Each mechanic is issued a pair of goggles which he keeps available for use when required. Monthly safety meetings help maintain interest in the safety program.

A large tool locker in the instrument shop includes such special items as socket wrench sets, pneumatic drill motor, electric drill motor, pneumatic impact wrenches for valve work, saw gun for panel board cut-outs, rotary saw for circular cut-outs, tubing tools, and many other items commonly used at most refineries. Replacement parts for equipment are stocked in the refinery storehouse with only enough parts kept in our shop to handle current needs.

Orifice Sections

All orifice sections or primary elements receive special attention from the instrument department personnel. There are a number of special points that must be observed and followed if satisfactory results are to be obtained from this type of metering equipment. These key points are as follows:

1. Length of straight pipe preceding and following the orifice must be carefully checked. The distances must conform to ASME requirements.

2. The line must be of uniform bore and free of burrs or roughness. Pipe must extend through flanges so there will be no recess between the face of the orifice plate and the end of the pipe. If weld neck flanges are used, the inside of the orifice section must be of uniform bore and free of welding burrs or roughness.

3. Orifice taps for liquid measurement are always made on the side of the line or flange. Taps for gas, air, or vapor measurements extend vertically from the line or flange.

4. The size of the tap may be within the limits of ¼ in. minimum to ¾ in. maximum, depending upon line size and service.

5. The pressure tap must be absolutely flush with the conduit wall and free of burrs or roughness. Microscopic burrs on the hole edge of an orifice tap have been known to produce errors as high as 4%.

6. Location of taps: Flange taps are to be 1 in. up and 1 in. down stream with respect to the face of the orifice plate. Pipe taps should be approximately 2½ pipe diameters preceding the plate and 8 pipe diameters following the plate. Vena contracta taps require that the up stream connection be one pipe diameter and the down stream connection be ½ pipe diameter from the face of the orifice plate. This varies slightly with the ratio of pipe to orifice diameters, generally referred to as Beta, or $d:D$ ratio.

7. The $d:D$ ratio should not be permitted to exceed 0.70 for best results.

8. Orifice plates must be very carefully centered in the line to produce correct pressure readings. The plate should be made to fit the bolt circle of the flanges with which it will be used.

9. Orifice edges must be square and sharp. Plate thickness must not be greater than 1/30 of the pipe diameter. On line sizes under 4 in., the plates are counterbored to the required thickness. The conventional beveled edge has been eliminated in favor of the square edge plate because of the error occasionally made by pipefitters during installation—inserting the plate with the beveled edge on the upstream side.

10. The orifice I. D. must be carefully checked during inspections and the handle marked to indicate the last calipered size.

11. Connecting lines between the orifice sections and the meter must not be less than ½ in. size. They should be graded at least 1 in./ft. to eliminate vapor or condensate traps.

12. In the measurement of light hydrocarbons, it is important to locate the orifice section at a point lower than the level of the material flowing from a drum or vessel. More volatile materials tend to flash when the orifice is installed above the mean liquid level.

Instrument Air Supply

Pneumatic control equipment must have an adequate supply of clean dry air for trouble-free operation. Extensive improvements in the air system have been made at Lockport to provide satisfactory instrument air supply.

Each operating unit in the refinery is equipped with air scrubbing equipment and auxiliary air compressors to take over in case the plant air system fails. These compressors "float" in the air line and cut in automatically at a predetermined minimum pressure.

Instrument Calibration

All differential type and mercuryless or forced balance instruments are calibrated dry using single-column manometers of various ranges. Low pressure gages and pneumatic receivers are carefully checked by comparison with a precision mercury manometer calibrated in lbs. and tenths, psi.

The indicating pressure gages are checked by comparison with laboratory test gages. The four master gages span the range of 0-3000 psi. and are tested once daily against the dead weight tester. This latter piece of equipment is ordinarily kept under lock and key at all other times, to reduce possibility of its being worn or damaged. With over 3000 gages in the refinery to be maintained, this test panel method has proved fast and efficient.

Potentiometer recorders are quickly and conveniently checked with a series of coils of fixed values. In operation, we replace the cold junction coil in the recorder with one of these coils of predetermined value. These coils supplement the portable testing equipment and are used primarily for determining whether the fault lies in the instrument or its external circuit.

Among the numerous special items worthy of mention are the pH recorders and controllers, and the gas analyzing equipment.

The determination and control of pH is important wherever water or water solutions are used. The modern oil refinery is continually discovering new benefits and new economies through the utilization of pH instruments. The instrument men are instructed in the servicing of this equipment. A daily check is required, and at present the cells are checked with a buffer solution which is verified by the control laboratory.

Various types of gas analyzers are in continuous use throughout the plant. Among these are oxygen analyzers, CO_2 recorders, and hydrogen analyzers, all of which are serviced daily by the instrument department.

It is not possible to mention in this space all the worthwhile practices and operating methods used in our department, but it is hoped that those presented may contribute something toward the improvement of refinery instrumentation.

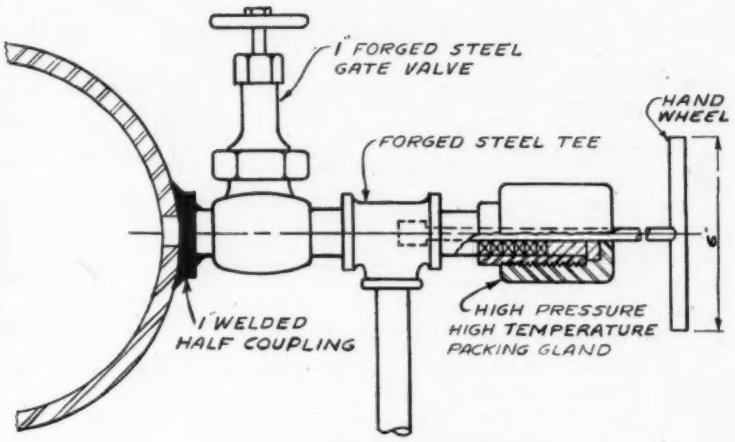
The standardization at our refinery of various items and of meter piping arrangements has done much toward improving departmental and equipment efficiency. Extra copies of drawings of these standards are kept on file. They are always available for all crafts assigned to do an instrument installation.

* * *

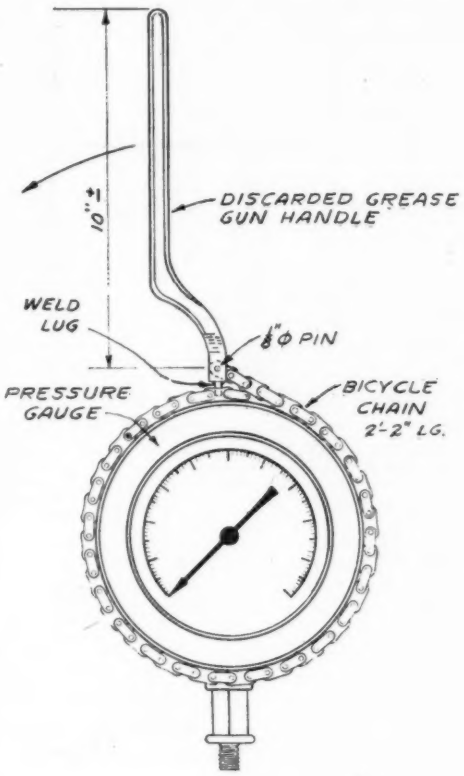
A pictorial representation of some of the typical installations and "gadgets" described in Mr. Larsen's paper has been selected by the editors of PETROLEUM PROCESSING and appears on the following pages. The material has been arranged for easy clipping by those readers who want to keep it in their instrument file.

Orifice Tap Cleanout

Detail of orifice or pressure tap cleanout device, designed for permanent installation in a line where frequent cleaning is necessary. In one installation in the flue gas system of a hydroforming unit, the equipment has been on stream over 2000 hours with no removal of instruments due to coke deposit. About once each shift, the operator pushes the plunger through the gate valve, removing any deposit which has collected in the valve and tee. When not in use, the plunger fits as a close seal at the outer end


1" FORGED STEEL GATE VALVE
HAND WHEEL
FORGED STEEL TEE
1" WELDED HALF COUPLING
HIGH PRESSURE HIGH TEMPERATURE PACKING GLAND
TO METER OR GAUGE

NOTE
FOR FLANGE TAPS USE ½" OR ¾" CONNECTIONS. SIZE OF TAP CONNECTION TO BE DETERMINED BY FLANGE WEIGHT.

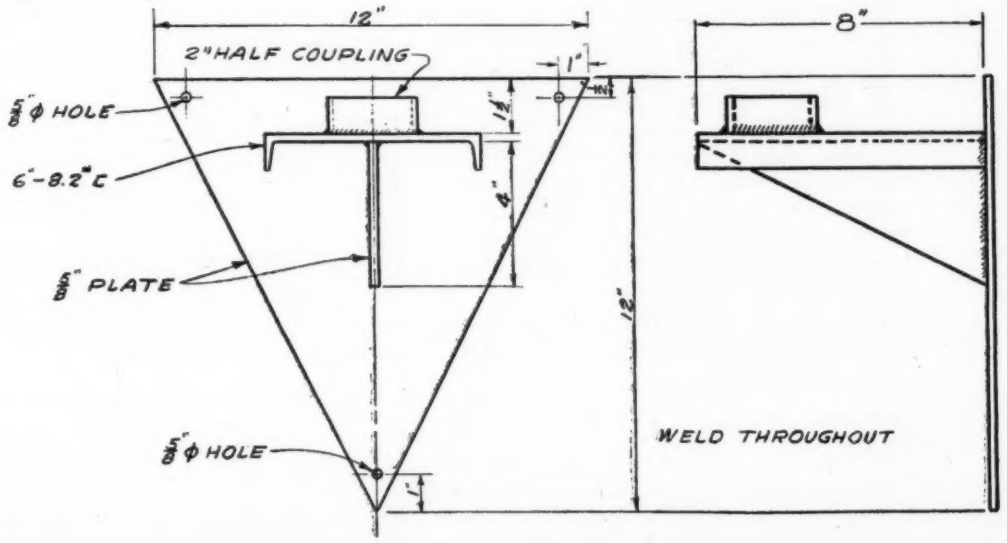


Spanner Wrench for Gage Covers

Special spanner wrench for removing threaded covers from pressure gages. These covers often stick and are quite difficult to remove. This simple wrench developed in the Lockport shop consists of a length of ordinary bicycle chain fastened to a discarded grease gun handle, and can be easily adjusted to accommodate all standard gage diameters

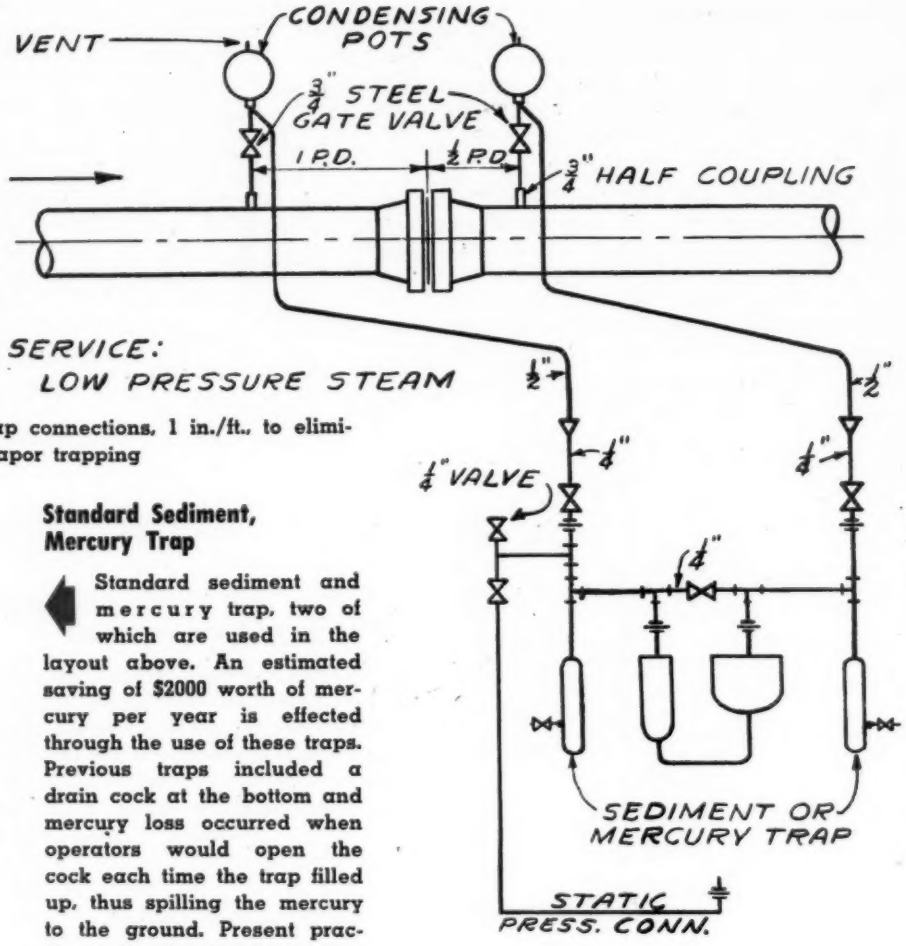
Standard Mounting Bracket

Standard wall bracket for meter and transmitter installations. These brackets are kept on hand as stock items in the instrument store room. They eliminate the former practice of assigning steel men or pipefitters to mount an instrument, using some bracket of their own individual design. The mounting height is a standard 42 in. from the floor to the top of the bracket. This procedure produces a uniform appearance and facilitates planning instrument layouts

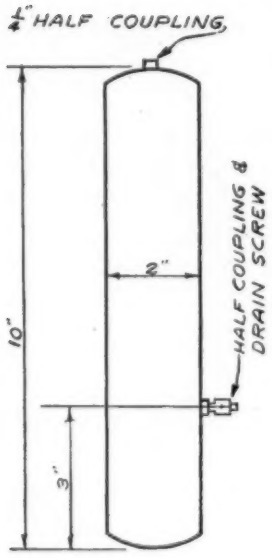


Low Pressure Steam Meter

Standard layout for installation of a low pressure steam meter. The two condensing pots above each pipe tap, details below, must be level and level with each other. Not indicated in the sketch, but adopted as standard practice is the slope of the pipe tap connections, 1 in./ft., to eliminate vapor trapping



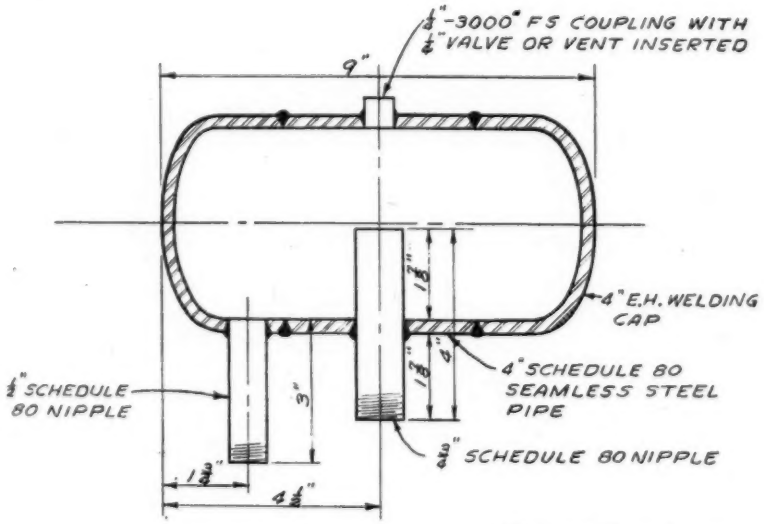
Standard Sediment, Mercury Trap

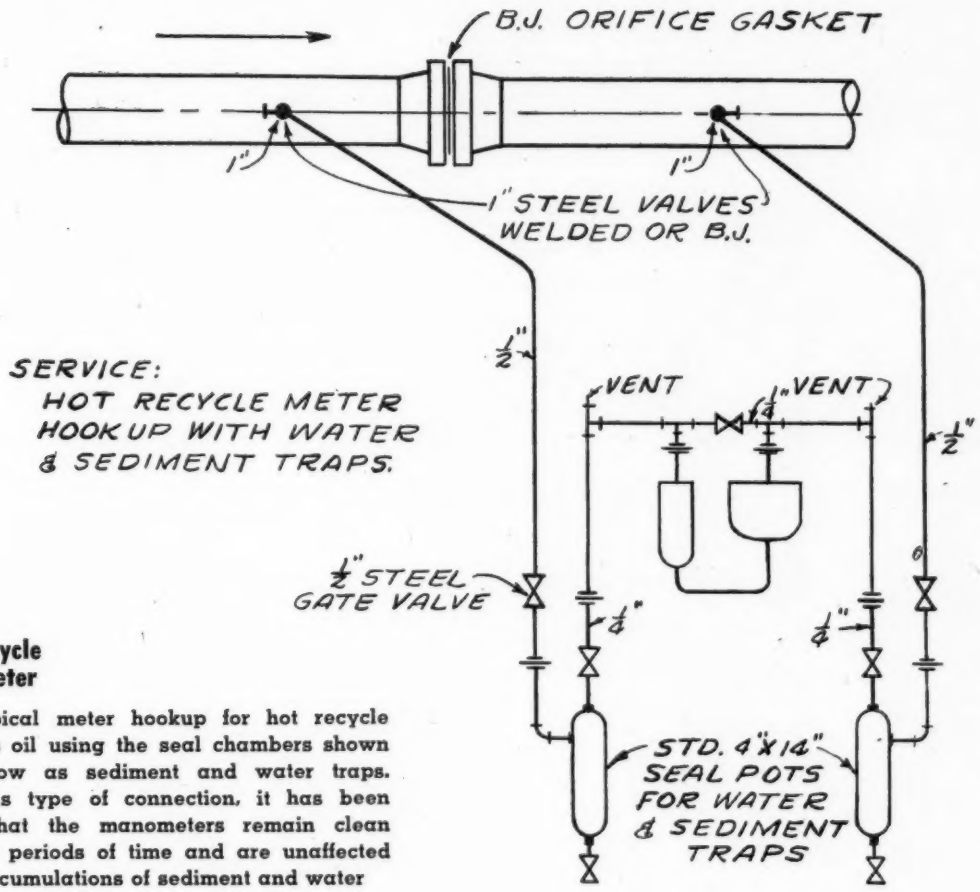


Standard sediment and mercury trap, two of which are used in the layout above. An estimated saving of $2000 worth of mercury per year is effected through the use of these traps. Previous traps included a drain cock at the bottom and mercury loss occurred when operators would open the cock each time the trap filled up, thus spilling the mercury to the ground. Present practice is for the instrument mechanic to replace a full trap with a clean, empty one. He then takes the full trap to the shop, drains the mercury, cleans and inspects the unit, and returns it to stock

Condensing Pot

Detail of condensing pot shown in layout above. Use of a ¾-in. drain line is said to provide sufficient capacity for drain back of condensate from the pot. In addition, its strength does away with the need for extra structural steel brackets to support the condenser, reducing the installation cost



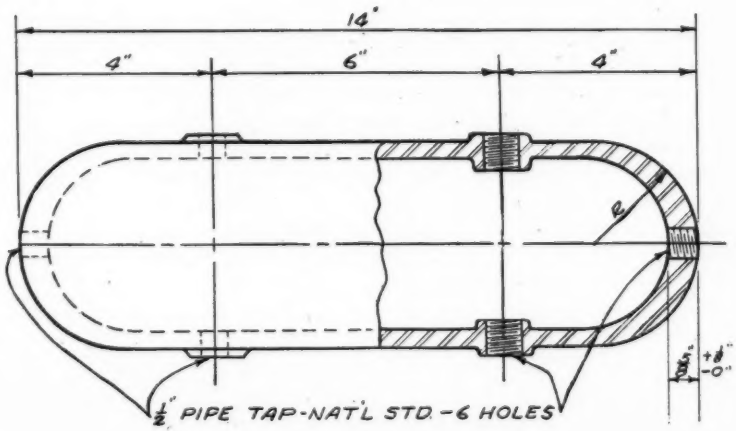


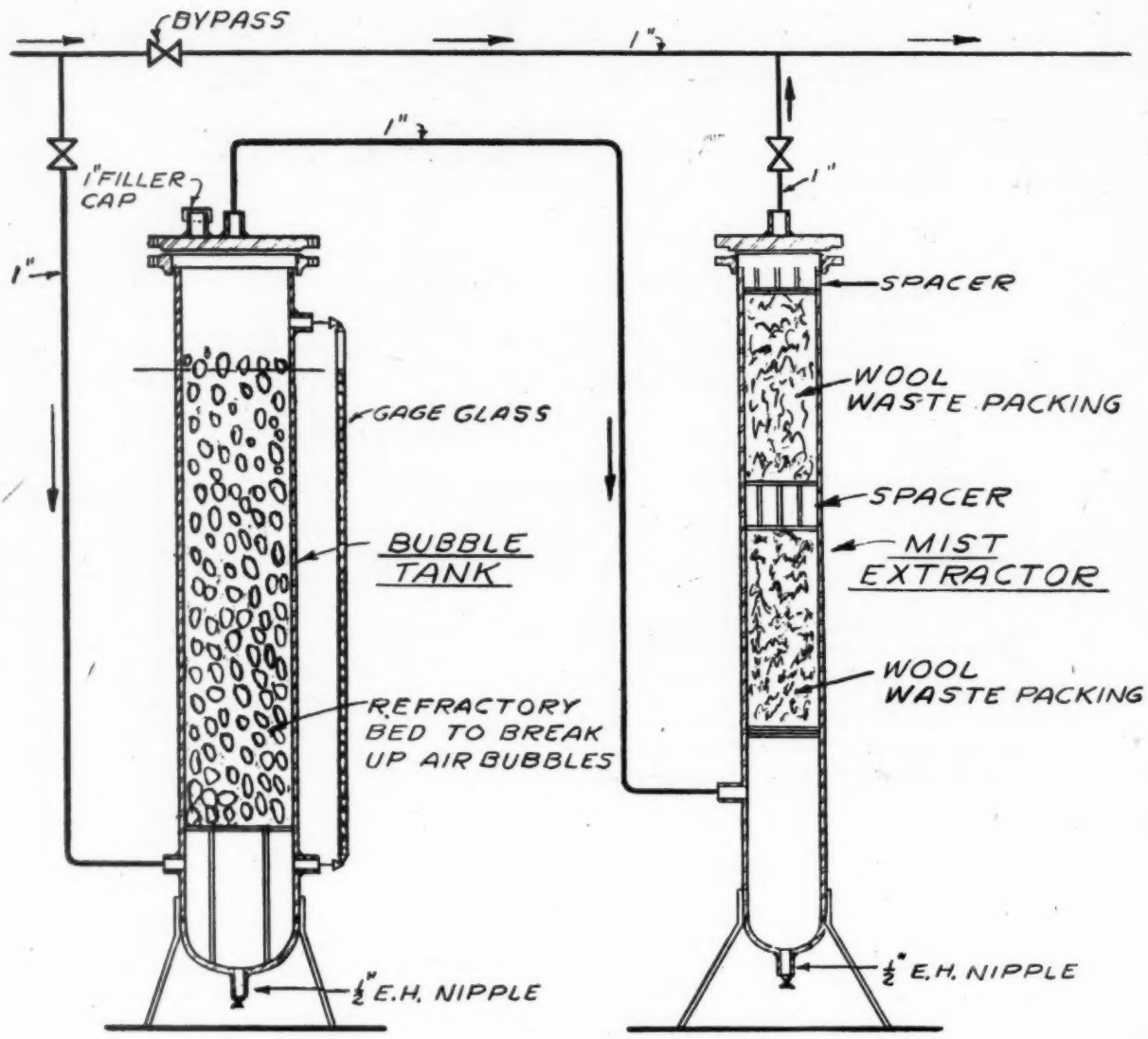
Hot Recycle Flow Meter

Typical meter hookup for hot recycle gas oil using the seal chambers shown below as sediment and water traps. With this type of connection, it has been found that the manometers remain clean for long periods of time and are unaffected by accumulations of sediment and water

Standard Seal Chamber

Detail of standard seal pot shown in layout above. Like the mercury traps shown on the preceding page, these traps are not made in The Texas Co. Lockport shop but are purchased from an outside source. They are made available in two types, depending upon service conditions which must be met. One is made from 4 in. Schedule 80 seamless steel pipe to meet a 1500 lb. hydrostatic test; and the other from 4 in. Schedule 160 seamless steel pipe to meet a 2000 lb. hydrostatic test. Both are made in one piece, the ends spun spherical and welded shut from spinning operation. The bosses are spun into the side





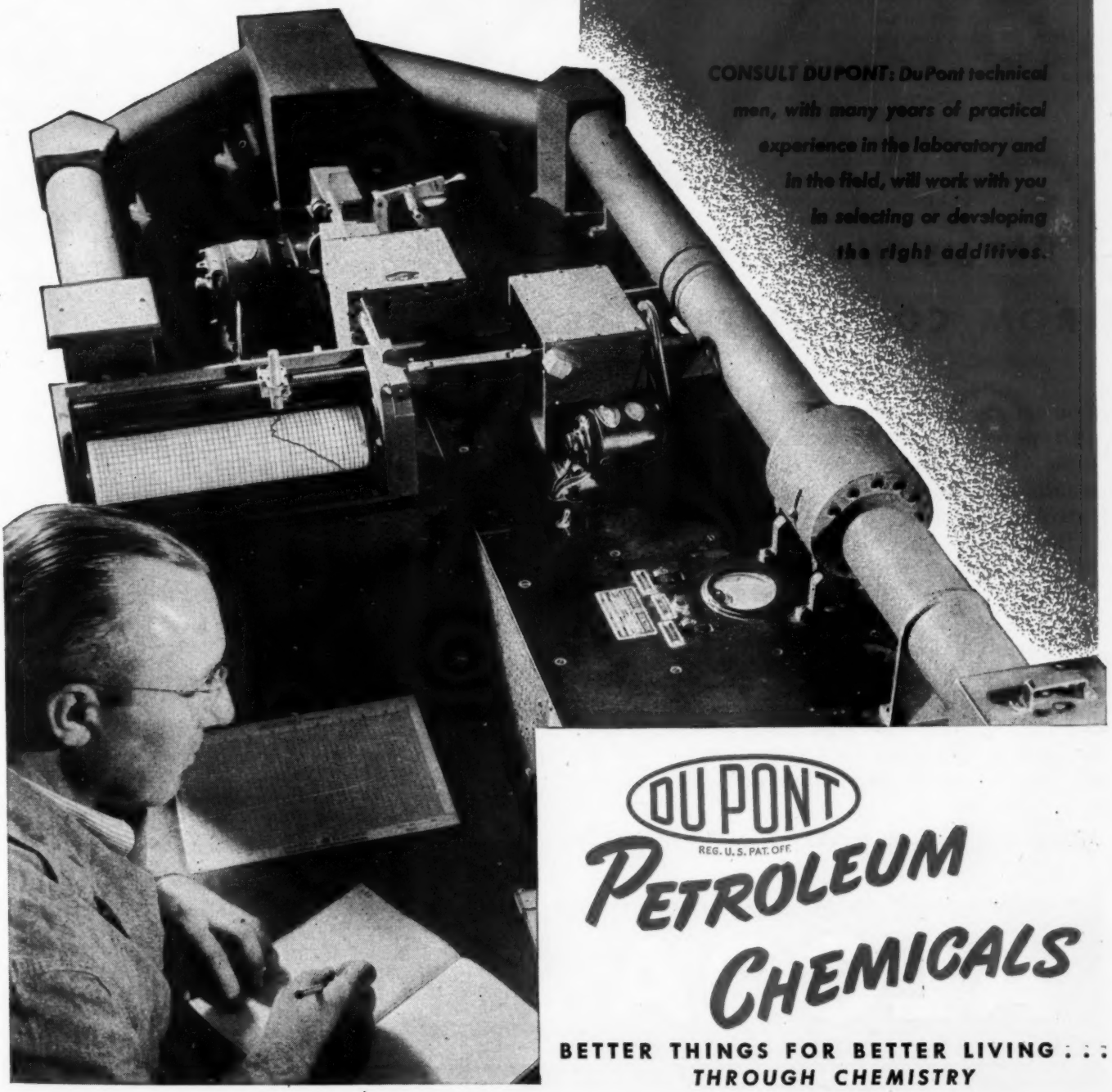
Instrument Air Cleaning Unit

Schematic diagram of an inexpensive and efficient shop-built air cleaning system. Constructed of 12-in. diameter pipe, with each tank approximately 6 ft. in height, this cleaner will handle the air for about 20 control instruments. Five of these units serve the instruments in the cracking plant at Lockport. The refractory bed in the bubble tank is made up of Raschig rings; the bubble tank liquid is transformer oil. The entire unit is cleaned out every six months. This cleaning unit is not a new idea, but was built during the war years when it was almost impossible to obtain a commercial type cleaner. In new installations commercial units will be used. However, the war-time, plant-built models are still capable of many years' service and will not be replaced

This recording spectrophotometer is one of the few such instruments in the country. It tells the complete story of a color—its strength, shade, and brightness —in a matter of minutes . . . and in writing.

Du Pont Oil Colors are carefully standardized by means of this instrument so that the refiner is assured of uniform tinctorial properties.

In addition to its use in controlling the quality of established products, it is valuable in determining the light transmission of unidentified dyes at each wave length in the visible spectrum—an essential operation in matching colors. *E. I. du Pont de Nemours & Co. (Inc.), Petroleum Chemicals Div., Wilmington 98, Del.*



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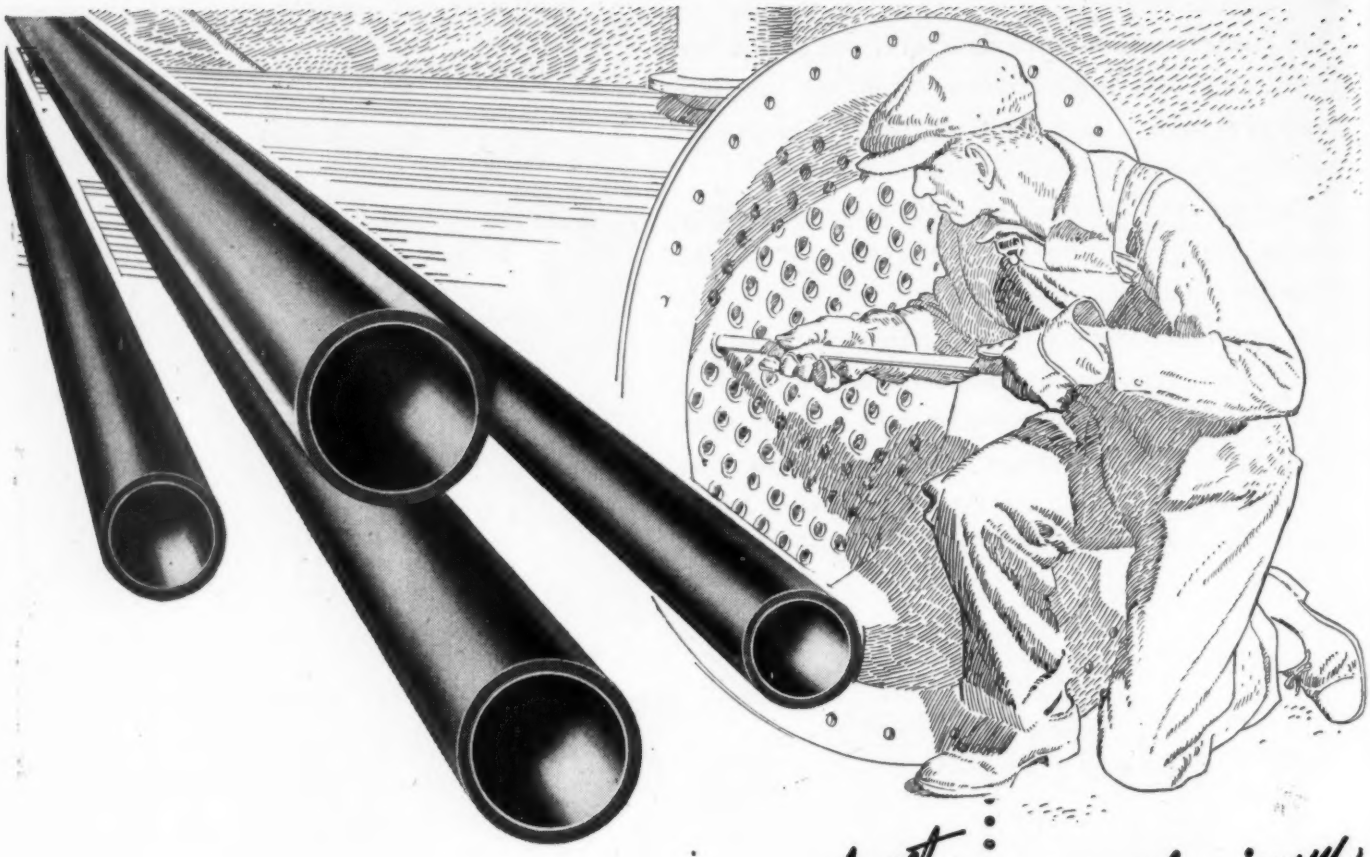

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PLANT PRACTICES

Practical Tips and Ideas for Improving Refinery Operations

Maintenance Men Lick Lumber Shortage, Build Movable Scaffold from Scrap Pipe

BECAUSE of the shortage of lumber for scaffolding, many refineries have found tank painting, insulating and reinsulating jobs a tough proposition in recent months. Shell Oil Co.'s Wood River refinery encountered this problem when plans were made to insulate a 15,000-bbl. tank, 40 ft. high and 60 ft. in diameter.

Ordinarily, a wooden scaffold all around the tank would have been the answer. Joint efforts of the plant boilermaker foreman and the engineering department resulted in the movable scaffold shown in the photograph. The unit allows four men to work simultaneously in safety and comfort, and it can be moved by one man.

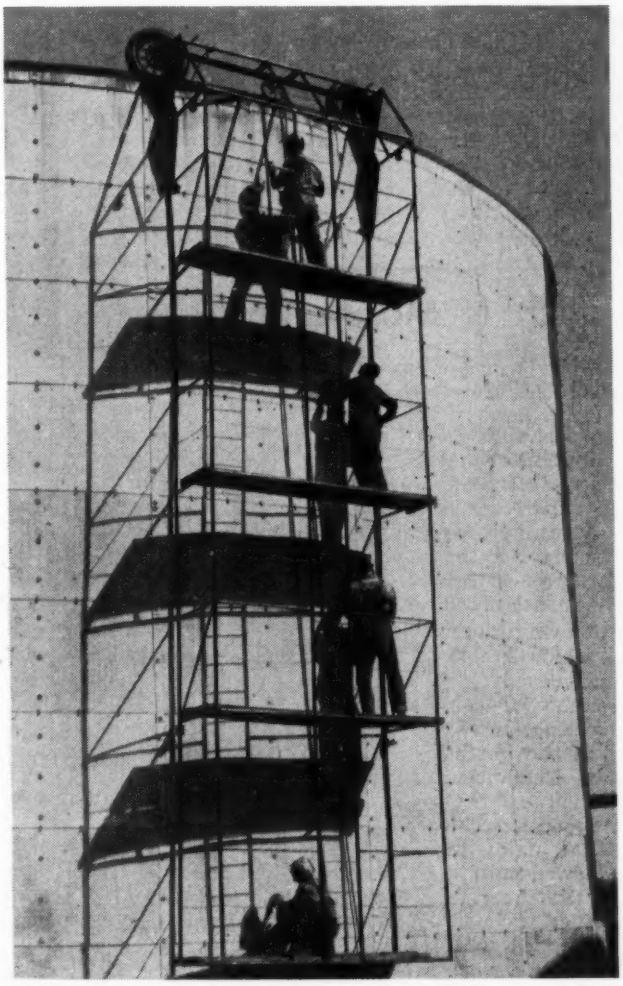
The all-steel scaffold is fabricated from salvaged 1-in. and 2-in. tubing. The four working levels are 8 x 8 x 3 ft., floored with movable 2 x 12's, and the scaffold is 8 ft. wide. Its height is 36 ft., of which approximately 35 ft. is actual working height and some 14 in. is above the top edge of the tank as supports.

Top support is by two automobile front-wheel assemblies mounting 6.00 x 16 tires, the spindles of which are mounted on the top piece of the scaffold with sufficient toe-in or camber to tend to follow a circular path, thereby keeping the scaffold against the side of the tank.

Lines are anchored to the center of the tank roof to offset any possibility for the wheels to leave the roof. The scaffold bears on the tank at the edge of the roof and the top ring. A pair of rollers at the latter point hold the scaffold an inch or so away from the tank wall.

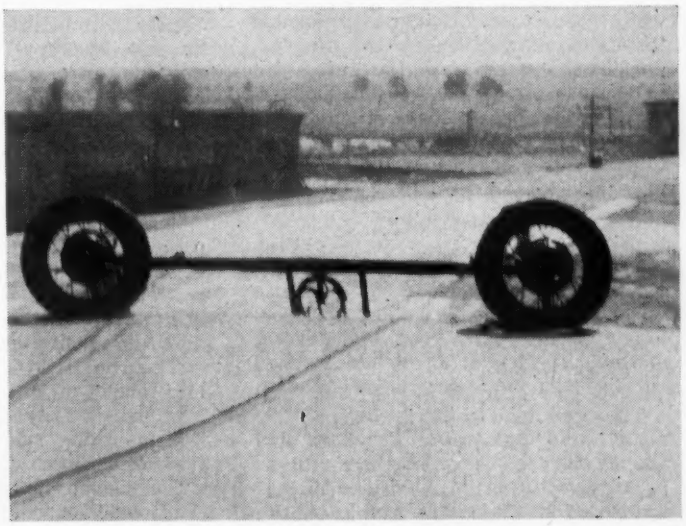
This scaffold is also used for sandblasting, painting and caulking where it works as well and eliminates the need for workmen to swing on stages. A rope and pulley, with which the scaffold is equipped, makes it necessary for the painter to carry only the usual brush and scraper in his pocket. The man nearest the ground can be given the job of replenishing the paint supply for the others, and a linetender for each painter is not required.

The company plans to make the next scaffold from aluminum rather than steel. Some redesigning is also contemplated to permit the height to be varied for different sized tanks and adapting it for both cone and floating roof tanks.



LEFT—All steel, movable scaffold used by Shell Oil Co. at Wood River refinery when 40 ft. high tanks need insulating or painting

BELOW—Top view of movable scaffold, showing detail of automobile wheel mounting. A line secured to the center of the tank roof prevents scaffold from rolling over the edge



Clean Out Condenser Stacks in 8 Hours With Low Labor Cost at Sun Refinery

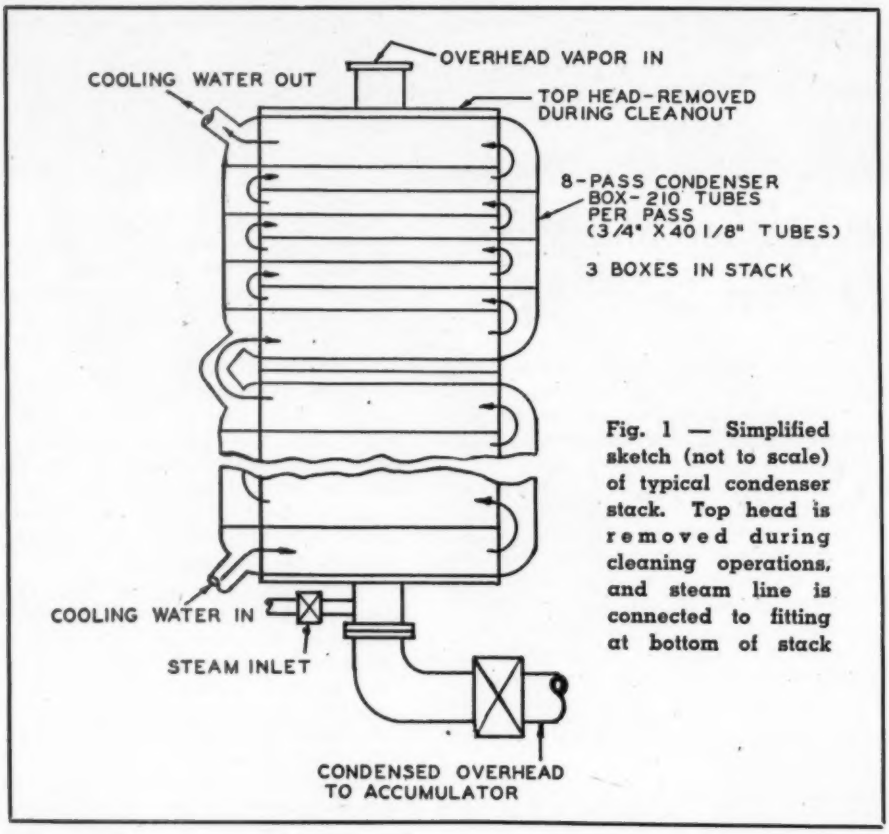

OVERHEAD VAPOR IN
COOLING WATER OUT
TOP HEAD-REMOVED DURING CLEANOUT
8-PASS CONDENSER BOX-210 TUBES PER PASS (3/4" X 40 1/8" TUBES)
3 BOXES IN STACK
COOLING WATER IN
STEAM INLET
CONDENSED OVERHEAD TO ACCUMULATOR

Fig. 1 — Simplified sketch (not to scale) of typical condenser stack. Top head is removed during cleaning operations, and steam line is connected to fitting at bottom of stack

CLEAN outs of condenser stacks on crude topping and thermal cracking units at Sun Oil Co.'s Marcus Hook refinery require only 6 to 8 hours time and virtually no labor through the installation of one fitting and the use of a steam and oakite treatment.

Each stack consists of three identical condenser boxes; as shown in Fig. 1. Each box contains 1680 horizontal ¾ in. diam. by 40 in. long cooling tubes, arranged with side baffles so as to produce 8 passes of water per box. Coolant enters at the bottom and leaves at the top. Overhead vapors from the unit enter at the top of the box and pass downward among the tubes. Condensate drains off from the bottom. Highest temperatures occur near the top, and it is at that point that the greatest amount of coke sludge accumulates.

Because the sludge is an oil-carbon combination, ordinary hot water or steam treatment accomplishes little in removal. The close clearance between tubes rules out mechanical methods. The solution as worked out by Sun engineers is as follows:

The top head of the stack is removed and 200 gals. of water and about 160 lbs. of oakite are run in (a 10% solution). A steam line is connected at the extra nipple on the bottom draw-off, and 100 psi. steam is bubbled through for about 6 hours. The oakite treatment dissolves the oil in the sludge, and the coke crumbles and can be easily washed away.

Develop Rapid Checking Method For Infra Red Spectrometers

A RAPID method for determining the linearity of the amplifying and optical system of a recording infrared spectrometer (Perkin-Aimer type) has been developed by Glyn R. Taylor, a laboratory employee of Humble Oil & Refining Co., Baytown, Texas.

Requiring five minutes or less and no specialized equipment or training, this method saves many man-hours per month by assuring that data are from a reliable, linear galvanometer, eliminates rechecks and additional computations from this cause.

Linearity of the galvanometer is determined as follows:

The instrument is set at any convenient wavelength of light, and the slit width adjusted to provide approximately 100 mm of galvanometer deflection without anything inserted in the light path.

The zero-line on the recording galvanometer is moved 15 to 20 mm at a time across the scale. The galvanometer deflection is observed each time; five intervals generally are sufficient. For re-check the zero-line is moved back across the scale, again recording deflections.

If the galvanometer is linear, deflections of the same magnitude should be recorded, regardless of the zero-line position.

Principle of the test is that, if the system is linear, the same galvanometer deflection should be obtained for the same amount of light admitted to the system regardless of zero setting.

Shop Workbench Is Mobile but Rugged

By GEORGE D. RHOADS, Ass't. Superintendent
Trinity Gas Corp., Hebbronville, Texas

IT is almost always desirable to have a good, ruggedly-built work bench in the center of a maintenance shop. However, the same space could often be used to advantage for heavy work right on the floor if the work bench were not there. Most work benches must be constructed solidly enough so that moving them becomes a major operation.

Trinity Gas Corp. has solved this problem at its Hebbronville, Texas, plant by installing an easily-moved work table. As shown in the photograph, the table may be dismantled and removed quickly.

The top is ½-in. steel plate. The legs are 2-in. pipe with a union in each leg. They are screwed into a 2-in. coupling sunk into the concrete floor flush with the surface.

The bench itself is equipped with the needed accessories, a pipe vise, pipe rest, and the like. One corner has 4 holes drilled and tapped for bolts that may be used to level a small base where level work is a "must."

Trinity Gas Corp.'s movable work bench

Pressure on Fuel Oil Line Controlled Through Air-operated Fluid Drive Unit

By ALFRED KRIEG, Supervisor, Instrument Department
Socony-Vacuum Oil Co. Inc., Paulsboro (N.J.) Refinery

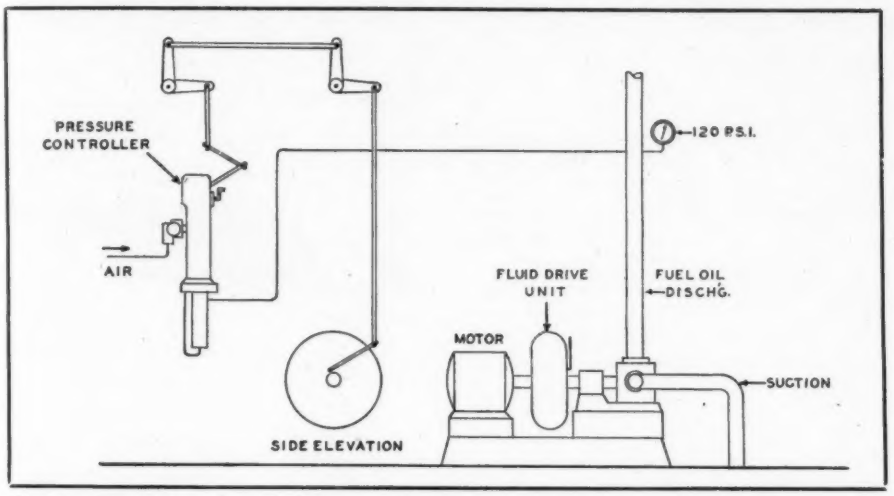


Fig. 2—Variations in discharge pressure are transmitted through air-operated pressure controller and lever arrangement to a scoop tube in the fluid drive unit, thereby controlling the speed of the pump

RECENT alterations and additions to the Power Department equipment at Socony-Vacuum's Paulsboro refinery were designed to increase the steam and power output. One pumping unit included in the new equipment consisted of a screw type pump driven by a 25 hp. electric motor. The pump and motor were coupled by a hydraulic coupling, or fluid drive unit, to supply fuel oil to the burners of the boilers.

The application involved required instrumentation for maintaining a constant pressure on the fuel oil line, and appeared to be a simple problem. The first suggestion, to use electrically operated speed control, would have required the conversion of pressure impulses into electrical impulses, entailing considerable expense.

Power requirements were beyond the range of the conventional diaphragm motor operated lever and would require considerable space for installation.

After careful consideration had been given to various types and methods for control, the system as shown in Fig. 2, was selected and installed.

The control unit consists essentially of a power cylinder, connected with a four-way pneumatically operated pilot valve. The fluid under control is piped to one side of a spring loaded bellows. The bellows motion operates the four-way valve which controls the flow of air to and from the power cylinder of the controller. Any motion of the piston is transmitted through the link and lever arrangement to operate the adjusting lever of the hydraulic coupling unit, controlling the pump speed and thereby maintaining a constant pressure on the burner fuel oil line.

The hydraulic coupling being controlled is similar to the fluid drive on late model motor cars, and consists of an impeller and runner and the housing or cover.

The radially vaned impeller, keyed to the driver shaft, is set facing the similarly vaned runner keyed to the driven shaft. In a sense, the runner sets within the impeller with both units enclosed, the enclosure being filled with a light turbine oil.

When the impeller is rotated at a high speed, the centrifugal force throws the oil outward between the radial vanes to the inside periphery of the coupling. The oil, following the curved contour of the impeller, flows directly into the vaned runner and back to the center to complete the circuit. The result of this continuous flow is a highly efficient method of power transmission by fluid motion.

The complete control coupling as installed includes the previously mentioned parts plus a rotating oil reservoir or casing, the manifold and an adjustable scoop tube.

The outer casing of the fluid drive unit acts as an oil reservoir and receives the oil not required in the circuit to transmit power.

The scoop tube is mounted eccentrically on the stationary manifold and pivoted to swing through an arc of 70°. When rotating, the oil is held by centrifugal force in an annular ring against the outer rim of the casing. The scoop tube may be adjusted between full dip and retracted positions by the lever, and will pick up a quantity of oil, depending on the position from the annular ring, carry it through the cored openings of the manifold and into the circuit to transmit power.

The lever and linkage arrangement is connected to this scoop tube adjustment lever actuated through the controller by the pressure impulses from the line.

Flange Spreaders Simplify Job Of Maintaining Refinery Piping

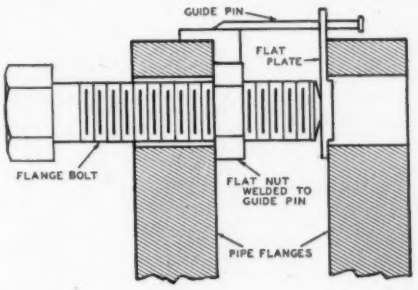


Fig. 3—Simple tool for separating large pipe flanges

INSERTION of blind spacers between pipe flanges is now done in about half the time required previously and with increased safety through the use of simple, shop-made "flange-spreaders" at the Torrance, Calif., refinery of General Petroleum Corp.

As shown in Fig. 3, the device consists of a flat nut threaded to fit the flange bolt. A guide pin is welded to the nut and, in turn, passes through a small hole in a flat steel plate. The plate is large enough to cover a flange hole and rest firmly against the inner surface of the flange.

The combined thicknesses of flat nut and plate must be less than the normal distance between flanges. Two spreaders are used in each operation. Previously a steel wedge was used for separating flanges. The method was hazardous as well as time-consuming. Wedges occasionally slipped out and injured the workman.

IDEAS—Wanted!

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Plant Practices Editor
PETROLEUM PROCESSING
1213 West Third Street
Cleveland 13, Ohio

It's A Hardy Fire That Could Even Start in *Utopia* Plant

By FRANK J. SLUZE, Safety Director
Ashland Oil & Refining Co.

Use of fireproof materials, strategically placed extinguishing equipment, and provision for quick "dropout" in all processing units means absence of fires in our mythical refinery—and would serve equally well in real plants.

Utopia Refinery's fire protection system is thorough. Our inspection, begun in the last installment in this series (Sept., pg. 662), will now be resumed.

On our way around the plant we observe several open faced boxes. Closer inspection discloses that each box contains a fog nozzle. Placing these nozzles at strategic and conspicuous locations is a good idea. Should an emergency warrant the use of such equipment, fire brigade members will know where to find one.

Let's inspect that pump room over there. We notice fire extinguishers hung on brackets just inside the door. Looking about, we find a steam smothering line has been installed. The control valve wheel is situated on the outside of the building—a good precaution. Steam can be poured into the room without requiring a man to enter. Chains controlling the doors and windows are equipped with fusible links set to melt between 110 and 130° F., thus closing all openings automatically should a fire break out.

Along the loading racks we see fire blankets enclosed in metal cases at regular intervals. The foreman explains that they are to be used in smothering tank car dome fires.

Inspection around the cracking plant discloses a number of monitors. These units are turret nozzles, or deluges, and are placed at strategic locations. An operator can aim them at a fire on adjoining equipment, turn on the water, and then retreat to a place of safety. In addition to these units, we are told that several have been mounted on trucks along with 200 ft. of hose. These wheeled monitors can be rolled into place, hose coupled to an adjacent fire hydrant, nozzle aimed, stream turned on, and again the operator can move off to a safe place.

Traps—but not like this one—are placed in catch basins to prevent fire from traveling through sewer lines

Fireproofing Also Necessary

While inspecting other parts of the operating area our attention is called to another precautionary measure—fireproofing stanchions and tower skirts by enclosing them with brick, concrete, tile, or insulation. This prevents heat from coming into direct contact with the metal and thus minimizes chances for collapse.

The operating foreman calls our attention to another safety measure—centrally-located, hydraulically-motivated, dropout lines. He shows us a board containing a number of levers. In case of emergency, such as fire, these levers are thrown into dropout position, emptying the towers or heaters in minimum time.

Looking overhead, we notice that, where possible, towers are interconnected with a cat-walk. This permits any person marooned above the fire to reach the farthest tower and then slide to the ground on the safety pole or line.

The operating foreman also points out the remotely-controlled stack dampers which may be used to cut off the draft of a furnace or heater, in case of a tube rupture for example.

He calls our attention next to the fact that brass valves are forbidden on any product line, and that cast iron valves are not permitted on any line where the temperature exceeds 450° F. This is a precaution against valve failure and the escape of oil which would provide additional fuel in the event of fire.

We notice a sign reading, "Caution—Purge furnace with steam before firing." Gas could accumulate and cause a nasty flash while a furnace was being reignited, hence the reason for purging with steam.

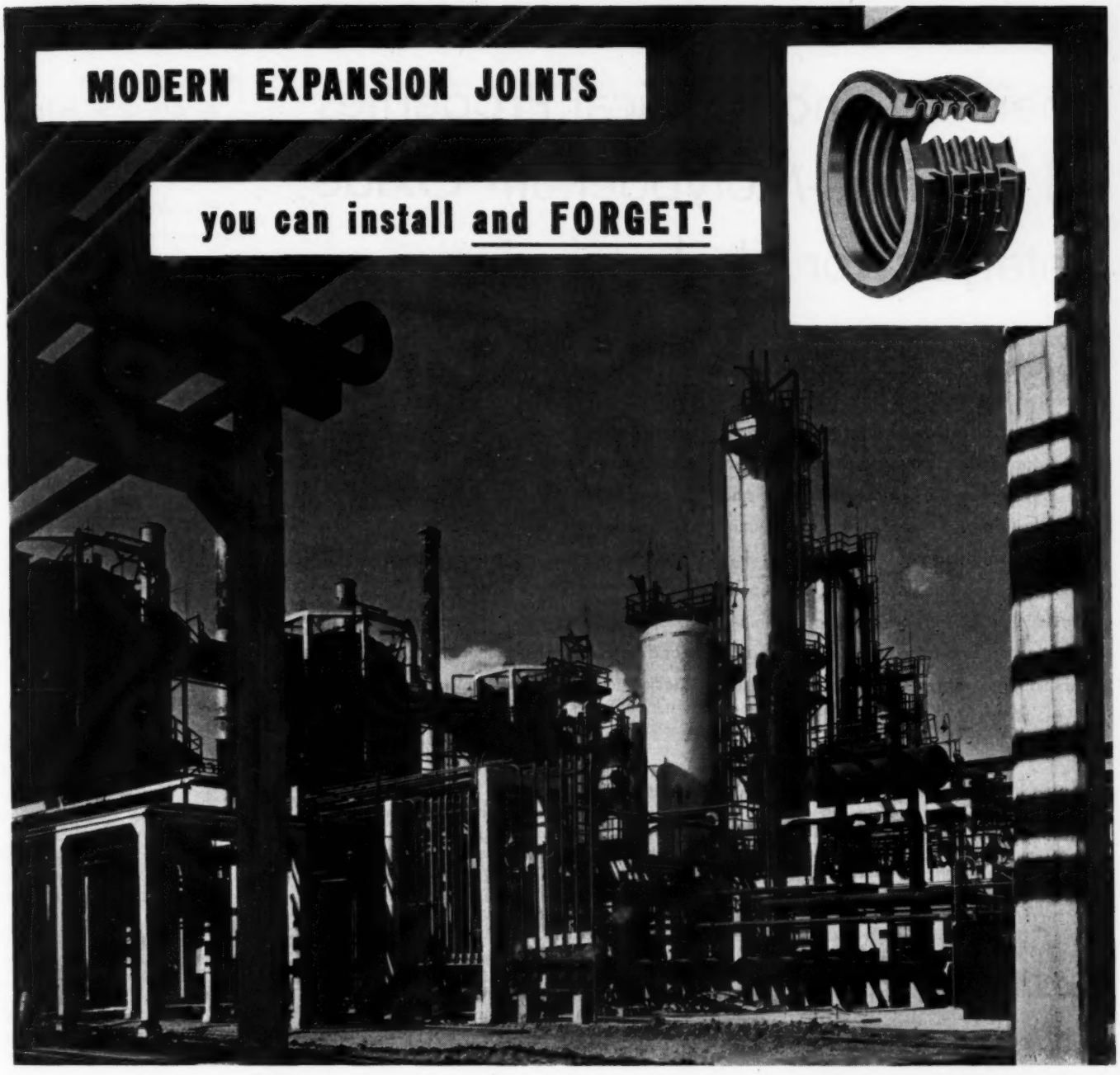
If trapped atop refinery towers, the Utopian can reach the ground safely and swiftly by means of slide wires

Inquiring about sewers, we are told that traps have been installed in the different manholes or catch basins so as to break the continuity of fuel flow in case of ignition of any one sewer. An additional safeguard has been provided by installing permanent steam smothering lines in all sewer manholes and catch basins.

Here in the operating area we again notice fire blankets in a metal case with a rope loop protruding. These are for saving personnel in case of clothing fires. The workman need only put his arm through the loop and turn around. That action pulls the blanket from the case and wraps it around the body.

Over at that furnace we see a box containing sand. We learn that these sand boxes are used to extinguish torches used in igniting a furnace.

Standing pools of oil are strictly forbidden and employes are instructed to clean them up or drain them off promptly. After this trip around the plant checking up on prevention, precautions, and procedures, we can readily understand why fires are nonexistent at Utopia Refinery.



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AGENTS IN PRINCIPAL CITIES

PACKLESS CORRUGATED EXPANSION JOINTS

Chemical and Physical Properties Of Alumina-Molybdenum Oxide Catalysts for Hydroforming

By GLENN M. WEBB
M. A. SMITH
C. H. EHRHARDT

Riverside Laboratories
Universal Oil Products Co.

Through studies of the preparation of an alumina-molybdenum oxide catalyst for hydroforming operations valuable data were developed on the relative values of impregnated and precipitation forms of this catalyst. The conventional impregnated form was found to be of low activity, high in coke formation and to have poor catalytic and strength stability. Catalysts made by precipitating alumina hydrogel in the presence of ammonium molybdate, followed by drying, calcining and pelleting, were found to be high in activity, low in coke forming and to possess greatly improved stability. Life tests on both the impregnated and precipitated catalysts were made in units operated under conditions similar to commercial operations, with test data taken under a special technique developed for evaluating catalysts.

DURING the past two decades many spectacular advances have been made in the application of the art of catalysis to a wide variety of industrial processes. Certain phases of the subject have been covered well in the literature, particularly with respect to the general types of catalysts that may be used for different reactions and the engineering aspects of various processes. But very little has been said of the properties of specific catalysts either as regards the manner in which these properties may be changed or how they may influence the course of the reaction being catalyzed.

This is a lack that should be rectified, for the success or failure of a given process may depend on whether the catalyst may be made economically from technical rather than from very pure raw materials; whether it has the needed stability, or life, in use; or whether it is poisoned by the compounds to be processed or by contaminants therein. Other questions that frequently arise in industrial operations concern the choice of the optimum particle size, it being necessary to use a size consistent with the fluid dynamics of the process and one which is also acceptable from the process standpoint as regards activity and carbon deposition.

In view of this unsatisfying state of affairs it was thought that it would be desirable to publish some of the work which has been done at the Riverside Laboratories on the properties of catalysts, and to indicate how use may be made of this information in making better catalysts. For these purposes, a description will be given of work on the development of a highly active, mechanically strong, catalytically stable alumina-molybdenum oxide catalyst used in hydroforming wide boiling range straight-run gasolines for octane improvement, or for treating narrow range stocks for the manufacture of aromatics, such as toluene.

Since the hydroforming process has been described in several articles[3, 1, 6,5] it will not be repeated in detail. Suffice to say that it involves catalytic dehydrogenation of the stock in the presence of hydrogen. The principal reaction that occurs in hydroforming is the dehydrogenation of C_6 naphthenes to produce, for example, toluene from methylcyclohexane, or xylene from dimethylcyclohexane. In practice, some side reactions occur, and a small amount of coke is deposited on the catalyst; consequently, the catalyst must be regenerated periodically. This is accomplished by stopping the flow of hydrocarbons to the reactor, purging the vapors from the catalyst case with inert gas, and finally burning off the coke with flue gas which contains a small amount of oxygen.

This processing and regeneration is accompanied by alternate reduction and oxidation of the molybdenum oxide in the catalyst. Consequently, the catalyst is subjected to severe thermal shock during both processing and regeneration. During regeneration a burning wave progresses through the catalyst bed, causing the temperature of the catalyst to rise above normal by as much as 100°-200°C. At the same time the catalyst is oxidized. After the carbonaceous deposit is burned off, hydrogen-containing gas is admitted to purge the regeneration gases from the system and to reduce the catalyst. This reaction is exothermic, causing the catalyst to be heated again to an abnormally high temperature. These operations put severe strains on the catalyst and it is essential, if the process is to be economical, that an extremely stable catalyst be used.

The catalyst used in much of the early work contained about 9% of molybdenum oxide deposited on activated alumina granules or pellets. This was made by the simple procedure of impregnating the alumina base with a solution of the proper concentration of ammonium molybdate. After being soaked for a time, the excess liquid was drained off and the wet pellets were dried and calcined. This catalyst, as will be shown later, was deficient in several respects, the most important being its rapid loss of activity and of mechanical strength during use under processing and regenerating conditions.

As a result of these observations, a systematic study was made of the physical and catalytic properties of

Earlier Catalyst Articles

Previous articles in PETROLEUM PROCESSING describing the work in the UOP Research Laboratories are:

"Properties of Cracking Catalysts", by Glenn M. Webb and C. H. Ehrhardt, January, 1947, pg. 7.

"Particle Size Distribution of Cracking Catalysts," by Glenn M. Webb, July, 1947, pg. 497.

the impregnated-type catalysts in the hope that leads could be obtained as to how the catalyst could be improved, or a new one developed. It turned out that the impregnated catalyst could be improved by effecting certain changes in the preparative procedure. It was also discovered that use of precipitation techniques rather than impregnation of a dry alumina resulted in catalysts that were tremendously superior in regard to initial activity and especially stability to heat. Consequently, the precipitated catalyst was studied in detail in the laboratory, and a protracted life test of some ten months' duration was carried out.

It is the purpose of the present paper to describe the pertinent parts of this work in detail. For convenience, the paper has been divided into two sections:

Part I. Properties of Impregnated Catalysts.

Part II. Properties of Precipitated Catalysts.

TEST PROCEDURES

Before the data are presented, the test procedures used will be described briefly.

CATALYST ACTIVITY TESTS

Evaluation of experimental catalysts for activity, and the running of the life tests were made in units which could be operated under conditions similar to those encountered in commercial operations. The feed stock used was a straight-run Mid-Continent naphtha having the inspection properties given in Table 1. Operating conditions were as given in Table 2.

In the activity evaluation tests, all catalysts were run for two complete cycles in order to obtain enough product for inspection. The regenerations were made with 0.7 cu. ft. of air per hour. In these tests, the catalyst bed temperature was usually about 17° F. below the block temperature. Carbon on the spent catalyst was determined either by combustion methods on a sample of unregenerated catalyst removed from the unit after the end of the second process period, or by absorption in an ascarite train of the CO_2 formed during the regeneration period.

In the life tests, regeneration was carried out using nitrogen which contained 2% O_2. Carbon on the catalyst was determined by absorption of the CO_2 in an ascarite train.

Product inspection included the Engler distillation curve; toluene content referred to the charge, the Reid vapor pressure of the product, and the F-2 (ASTM) octane number.

TABLE 1—Properties of Feed Stock Used in Catalyst Evaluation Tests

(Straight Run Mid-Continent Naphtha)

Gravity, °API	52.7
Molecular Weight	132
Engler Distillation, °F.	
I.B.P.	221
10%	261
30%	288
50%	312
70%	336
90%	368
E.P.	415
Bromine No.	< 0.5
n_D^{20}	1.4273
PONA Analysis, % by weight	
Paraffins	61
Olefins	0
Naphthenes	26
Aromatics	13
Toluene, % by weight	2.0
F-2 octane rating	36.5

TABLE 2—Operating Conditions for Catalyst Evaluation and Life Tests

	Catalyst Evaluation Tests	Life Tests #1	Life Tests #2
Catalyst Volume, ml.	100	166	100
Furnace Block Temperature, °F.	932	977	950
Pressure, psig	100	200	100
Space Velocity, V/V/Hr.	0.5 or 1.0	0.60	0.50
Hydrogen Feed Rate, Mols. H_2/mol. Naphtha	4.5	3.5	3.0
Process Period Length at Plant Pressure, Hrs.	6	6	6
Hydrogen Purge Period at Plant Pressure, Min.	4	4.2	3
Time to Depressure to Zero lbs./in.² Gage, Min. psig, Min.	3	1.5	3
Nitrogen Purge at Atm. Pressure, Min.	3	1.9	3
Regeneration at Atm. Pressure, Hrs.	3	2.6	2.4
Nitrogen Purge at Atm. Pressure, Min.	4	7	3
Hydrogen Purge at Atm. Pressure, Min.	4	3	5
Pressure Buildup with H_2, Min.	1	1	1
Hydrogen Purge at Plant Pressure, Min.	2	2	...

CATALYST PROPERTIES

Structures

The crystalline structures of the catalyst were determined with a G.E., X.R.D. diffraction unit by the Debye-Scherrer powder method using CuKα radiation and a camera of 45 cm. circumference.

Surface Areas

These data were obtained by Dr. G. Jura and Dr. W. D. Harkins[4] of the University of Chicago by low tem-

THIS MONTH'S COVER

Making Catalyst for Testing at Riverside

Photo courtesy Universal Oil Products Co.

This month's cover picture shows a typical set-up of equipment used in the Riverside Laboratories of Universal Oil Products Co. for making experimental batches of catalysts to be used in test work.

In the picture, M. A. Smith, one of the authors of the accompanying article, is shown preparing highly active alumina. The procedure being followed consists of adding ammonium hydroxide to a solution of aluminum chloride to precipitate alumina gel. A rough measure of the end pH is gained by use of an indicator, which is being added with a medicine dropper. The end pH is determined accurately by means of the pH meter shown on the table. The final pH reached in such a preparation is important because it determines to a considerable extent the properties of the finished product.

After precipitation is complete, the slurry is transferred to the Buchner funnels on the table and separated from the mother liquor, followed by alternate re-slurrying in wash water and filtering to remove impurities.

After several washings, the cake is dried under carefully controlled conditions and, if desired, pelleted to the required size and shape.

In the background at the right are shown vertical tubular furnaces for carrying out the final treatment. At the far left on the table appears a flame photometer, used for determining the sodium content of catalytic material.

TABLE 3—Effect of Heat Treatment on Loss of Molybdenum Oxide, Strength, and Structure of Hydroforming Catalysts

		Properties After 6 Hrs. @ 1112° F.		Properties After 6 Hrs. @ 1472° F.				Properties After 6 Hrs. @ 1652° F.		Properties After 6 Hrs. @ 1832° F.	
Cat. No.	Type of Alumina	Crush Strength lbs.	% MoO_3 Wt. %	% MoO_3	Structure	Color	Crush Strength lbs.	Structure	Color	Structure	Color
1	$Al_2O_3 \cdot 3H_2O$	3.9	8.89	8.17	γAl_2O_3	White	7.7	αAl_2O_3	Blue	.....	Blue
2	$Al_2O_3 \cdot H_2O$	2.0	9.22	8.40	γAl_2O_3	White	2.3	αAl_2O_3	Blue	αAl_2O_3	Blue
3	γAl_2O_3	13.6	8.88	7.83	*	White	3.7	αAl_2O_3	Blue	αAl_2O_3	Blue
4	γAl_2O_3	4.5	3.29	3.31	αAl_2O_3 + *	Blue	1.9	αAl_2O_3	Blue	.....	Blue
5	γAl_2O_3	4.9	7.42	7.31	.....	....	2.2	αAl_2O_3	Blue	.....	Blue
6	γAl_2O_3	4.8	10.20	9.92	αAl_2O_3	Blue	2.5	αAl_2O_3	Blue	.....	Blue
7	γAl_2O_3	5.1	13.34	13.56	.....	....	2.6	αAl_2O_3	Blue	.....	Blue
8	$Al_2O_3 \cdot 3H_2O$ .2-.6μ	1.0	9.16	8.02	γAl_2O_3	White	2.1	*	White	αAl_2O_3	Blue
9	γAl_2O_3	..	8.22	7.89	γAl_2O_3	White	..	αAl_2O_3	Blue	αAl_2O_3	Blue
10	γAl_2O_3 (contain clay)	..	8.72	9.47	γAl_2O_3	White	..	*	White	αAl_2O_3 + ?	White

* Represents crystal growth, the nature of which is not known.

perature nitrogen adsorption method(2).

Crushing Strengths

The strengths of the pelleted catalysts were measured by determining the weight required to crush the pill. In making this test, the pill is placed on its side, and lead shot are made to fall at a steady rate into a cup attached to a flat ended plunger, the bottom end of which rests on the top side of the pill. When the pill is crushed, the flow of lead shot is automatically stopped. The cup, plunger, and lead shot are weighed and the total weight is termed the "peripheral crushing strength."

Density

Two densities are normally measured for each catalyst. The first, apparent bulk density, is determined by measuring the weight of pills contained in a 100 ml. container. The pills are allowed to fall slowly into a funnel held above the container. The second, piece density, is determined on about 25 ml. of catalyst by a mercury displacement method, and represents the average density of the individual pellets. If the tests are made carefully, it is usually found that the apparent bulk density is proportional to the piece density.

PART I

IMPREGNATED-TYPE CATALYSTS

Structure of Various Hydroforming Catalysts. Effect of Heat Treatment in Producing Changes in Structure.

ONE of the most important properties of a catalyst is its thermal stability. It has been our opinion that for successful commercial operation, a catalyst should show no tendency to change its crystalline form or composition when heated, say 200° F. above the highest temperature to which it will be exposed in large-scale regeneration operations. Hence, alumina molybdenum oxide hydroforming catalyst, which is usually regenerated at temperatures up to 1200° F., should be able to withstand temperatures of the order of 1400° F. without noticeable damage.

To determine whether hydroforming catalysts then available suffered any structural changes upon being heated to relatively high temperatures, a group of experimental catalysts and several commercial catalysts were calcined in air for six hours at 1112°, 1472°, 1652°, and 1832° F., and examined by X-ray diffraction methods. The samples calcined at 1112 and 1472° F. were analyzed for molybdenum oxide content.

It does not necessarily follow that changes in structure or composition observed as a result of heat treatment will be accompanied by a decline in the activity characteristics. However, on the basis of previous experience, such a decline would be expected, particularly for alumina base catalysts, since the most probable product, αAl_2O_3, is notoriously poor as a catalyst support.

Ten representative catalysts studied in this connection are listed in Table 3, along with some of their physical and chemical properties. Catalysts 1, 2, 3, and 8, prepared by impregnation of the alumina powder before forming, differ mainly in the structure of the alumina base at the time of impregnation. Gibbsite ($Al_2O_3 \cdot 3H_2O$) was used for No. 1; Boehmite ($Al_2O_3 \cdot H_2O$) for No. 2, and γAl_2O_3 for No. 3. No. 8 was prepared from Gibbsite of very small particle size (0.2-0.6μ). All were finally calcined at 1112° F.

Catalysts 4 to 7 and 9 and 10 were prepared by impregnation of preformed ⅛ in. by ⅛ in. cylindrical pills, with ammonium molybdate solution for the purpose of studying the effect of molybdenum oxide content. Catalyst 10 is interesting because it contained a small amount of clay.

RESULTS

Loss of Molybdenum Oxide

All of the catalysts listed were analyzed for molybdenum oxide content after both the 1112° and 1472° F. calcination. The fact that such a small fraction of the molybdenum oxide was lost when the catalyst is heated in air at 1472° F. was surprising, since it was believed that it would be fairly rapidly volatilized at that temperature. That it was not volatilized to any appreciable extent led to the thought that either a nonvolatile type of molybdenum oxide was formed as a result of the heat treatment, or that a complex between the molybdenum oxide and the alumina was produced. This point will be discussed in more detail presently.

Effect of Heat Treatment on Catalyst Strength and Structure

The crushing strength of the catalysts tended to decrease as a result of the heat treatment. When the initial crushing strength after 1112° F. was low, slight apparent increases in strength were measured, but it is doubtful whether small differences in the range of 1 to 2 pounds crushing strength have any significance. It was concluded from these and other experiments that calcination of alumina-molybdenum oxide catalyst at

TABLE 4—Effect of Molybdenum Oxide Concentration on Catalyst Activity

Molybdenum Oxide Content Wt. %	960° F.			990° F.			1020° F.		
	Wt. % Gasoline	Wt. % Carbon	O.N. F-2	Wt. % Gasoline	Wt. % Carbon	O.N. F-2	Wt. % Gasoline	Wt. % Carbon	O.N. F-2
Quartz (Blank)	...	...	...	...	...	...	89.7	0.0	49.3
0	...	...	...	87.7	0.01	54.9	82.6	0.02	60.7
3.2	87.1	0.31	74.8	78.0	0.70	81.5	70.8	0.76	84.8
7.2	77.5	0.80	82.0	71.8	0.93	85.9	67.2	1.27	89.5
10.2	78.4	1.32	81.7	73.8	1.05	85.1	66.8	1.75	88.1
13.3	78.3	1.37	82.1	71.4	1.61	84.3	65.8	1.93	87.8

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The answers to your Questions about Du Pont Tetraethyl Lead Compounds

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Although Du Pont has been manufacturing tetraethyl lead for a number of years it has not until now been engaged in the marketing of this important commodity. Beginning January 1, 1948, Du Pont will offer tetraethyl lead anti-knock compounds and services directly to refiners.

To meet the increasingly complex needs of the industry and to provide a broadened service for its customers, Du Pont has formed the Petroleum Chemicals Division. Through this Division, Du Pont will offer tetraethyl lead, oil dyes, gasoline antioxidants, lube oil additives, etc.

How long has Du Pont been manufacturing tetraethyl lead?

Starting in September, 1923, Du Pont produced its first commercial quantities of TEL. Up until two years ago, practically *all* of the TEL manufactured in this country was made by Du Pont. In the past 24 years, Du Pont has produced over two billion pounds of TEL compounds.

Under what name will Du Pont TEL be sold?

There will be two: Du Pont Tetraethyl Lead Compound—Motor Mix and Du Pont Tetraethyl Lead Compound—Aviation Mix.

Will the TEL compounds offered by Du Pont after January 1st differ from those now being made?

No.

What requirements must the refiner meet in order to use Du Pont Tetraethyl Lead Compounds?

Only those imposed by safety and health.

Must fuels containing Du Pont Tetraethyl Lead Compounds be dyed a specific color?

All fuels containing TEL compounds must be dyed a distinctive color, according to the requirements of the Surgeon General. However, the choice of color for gasolines in which Du Pont Tetraethyl Lead Compounds are used will be left entirely to the refiner. Dyes for coloring gasoline are manufactured and sold by Du Pont.

May present lead blending equipment be used?

Since this equipment is standard, it may be used for any tetraethyl lead compound. Replacement parts for existing equipment and new units will be available from Du Pont.

In what units will Du Pont TEL Compounds be sold?

They will be sold in standard containers and tank cars. Du Pont will have its own fleet of completely new tetraethyl lead tank cars.

What services will Du Pont provide?

Du Pont will offer comprehensive technical, operating and medical services.

What medical services will be available?

Du Pont's large staff of physicians and technicians, backed by many years of experience and supplemented by complete laboratory facilities, will be available to serve the industry. Their services will include the periodic examination of all personnel working with tetraethyl lead compounds. Advice on general health problems relating to the use of Du Pont additives will also be available.

What operating and safety services will Du Pont offer?

The extensive knowledge of operating procedures and safety in handling tetraethyl lead, gained by Du Pont during the past twenty-four years, will be made available to the refiner through a well-trained and experienced service organization with representatives in all major refinery centers. They will act in an advisory capacity on all problems associated with the handling of tetraethyl lead.

What services will be provided by the district laboratories?

District laboratories of the Petroleum Chemicals Division will be located at Houston, Texas; Wilmington, Delaware; Chicago, Illinois; Tulsa, Oklahoma; and El Monte, California. They will at all times provide helpful, cooperative service in the solution of specific problems.

Will Du Pont do fundamental combustion research?

In line with its well-established policy, Du Pont will engage in a broad program of fundamental research in this field. Extensive chemical research laboratories are available to carry on this work, and engineering and road testing laboratories are being established in order to investigate all phases of fuel utilization.

Will the petroleum industry benefit from Du Pont company-wide research?

The findings of any one of the many Du Pont laboratories throughout the country are available to the Petroleum Chemicals Division. From this interchange of advance knowledge comes a broad approach to any problem.

Does Du Pont produce other products for the refiner?

YES! In addition to additives for petroleum products, of which there are many, Du Pont produces hundreds of products which find direct or indirect uses in the production and refining of petroleum. Included are such things as chemicals, paint, neoprene, explosives and many others. Both as a major user of petroleum products and a supplier of chemicals for the petroleum industry, Du Pont has always been keenly interested in the growth and development of the industry and the betterment of its products.

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temperatures in the range of 1472° F. results in a loss of strength.

The X-ray data showed that all of the catalysts contained γAl_2O_3 after the 1112° F. heat treatment in air. In no case was the presence of any molybdenum oxide clearly demonstrated in these X-ray tests. This simply means that if the molybdenum oxide exists in the crystalline state in the catalyst its crystallite size is too small to be discovered by the X-ray method. All of the catalysts except No. 10, which contained clay, were similar in color; namely, white.

Not all of the catalysts calcined at 1472° F. were examined by X-ray diffraction. In those that were so examined, it was found that two crystalline components were present, namely, γAl_2O_3 and a component of undetermined crystalline nature.

When the catalysts were calcined at 1652° F., a large amount of crystal growth and change occurred in all of them. The most remarkable feature of the majority of the X-ray patterns was that they showed the presence of large amounts of αAl_2O_3. Those catalysts containing clay, or prepared from the finely divided alumina (.2—.6μ) showed considerably less crystal growth than the other samples.

The color of the catalysts seemed to correlate with their structures. The 1652° F. calcination caused all of the catalysts, with the same exceptions noted above, to turn blue. All of the catalysts which had become blue contained α alumina; those which had not changed color contained minor proportions of αAl_2O_3 and considerable quantities of the intermediate material, which had not yet been identified. The edges or corners of the pellets or granules in contact with the clay crucible, in which the samples were calcined, did not turn blue but remained white.

After the 1832° F. calcination, all of the samples except those which contained clay were blue in color and contained αAl_2O_3. The clay-containing samples also contained αAl_2O_3 but showed less general crystal growth than the others.

The unidentified crystalline component encountered was not identical with any of the material found in the transition of pure alumina from the γ to the α form upon heat treatment in the temperature range 1600-2000° F. Because of this fact it is thought that this material is a complex of alumina and molybdenum oxide out of which αAl_2O_3 subsequently separates, leaving the molybdenum oxide in the form of molybdenum blue.

As a result of these structural studies, it was concluded that the rate of change of crystalline form of the catalyst with temperature is controlled by the composition of the catalyst. Two types of catalyst which seemed to resist structural change were those such as No. 10 (containing some clay) and No. 8, made from very finely divided alumina. No. 10 also differed from the others in that the alumina base contained about 0.3% Cr_2O_3 as an impurity. It therefore appeared that any one of three items might be responsible for stabilizing these catalysts: Alumina particle size, presence of Cr_2O_3, presence of clay.*

As a result of speculation along these lines a series of catalysts was made in order to determine the effect of each of these factors. The catalysts were made in small quantity (about 100 gm.) and were not pelleted nor extruded. They were impregnated with ammonium molybdate solution in order to contain 9% MoO_3, dried at 300° F., and examined for color only after calcination at 1112° F., 1472° F., and 1652° F. Catalysts were made from various aluminas, with and without clay, and from alumina containing Cr_2O_3.

The results indicate that Cr_2O_3 has little or no stabilizing effect. It appears that particle size of the alumina does have an effect on the stability of hydroforming catalysts, increased stability being associated with small particle size. The data indicate that the presence of clay (Bentonite) has a definite stabilizing effect on catalysts of this type.

The effect of clay and other stabilizers has been investigated in much greater detail both structurally and from an activity standpoint, and will be discussed shortly.

*We wish to acknowledge the cooperation of the Standard Oil Co. of New Jersey, Louisiana Division, who described their own independent work on clay-stabilized hydroforming catalyst to us at the time we were doing the work herein described.

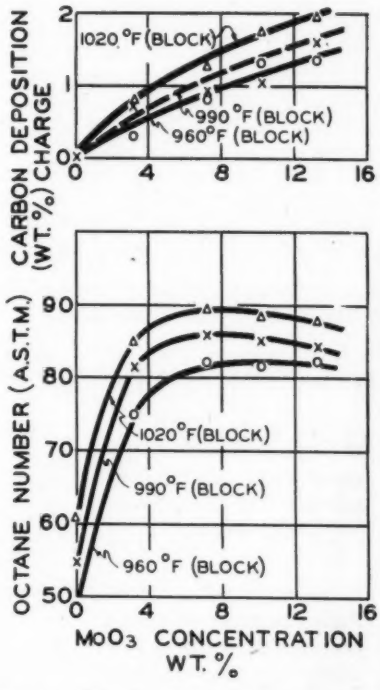


Fig. 1—Effect of molybdenum oxide concentration on octane number and carbon deposition at various temperatures (impregnated catalysts)

Effect of Molybdenum Oxide Concentration on Catalyst Activity and Coke Deposition

The determination of the optimum concentration of molybdenum oxide in the catalyst is important for a variety of reasons, the foremost of which is probably based on the considerations that the most desirable catalyst is the one which shows the highest activity and the lowest coke deposition, and that this combination of high activity and low carbon may only be achieved at a particular ratio of alumina to molybdenum oxide. Therefore, a series of tests was designed to yield information on the dependence of activity and carbon deposition with varying molybdenum oxide concentrations. It is realized that the optimum concentration of molybdenum oxide, if such exists, may and probably will depend upon the method of preparation of the catalyst.

The present series of catalysts (No. 4, 5, 6, and 7, Table 3) was prepared by the impregnation of preformed ⅛-in. alumina pills with ammonium molybdate solutions of proper concentration. This method was chosen because of its simplicity, and since it allows the molybdenum oxide concentration to be changed without at the same time changing other variables.

The catalysts tested contained 0, 3.2, 7.2, 10.2, and 13.3% MoO_3 by weight. Tests were made at 960, 990 and 1020° F. (block temperature). The results are shown in Table 4 and Fig. 1.

It will be observed in Fig. 1 that from the standpoint of octane number of the product, an optimum concentration of molybdenum oxide appears to exist and is in the region of 6 to 10% MoO_3. The carbon data also given in Fig. 1 show that carbon formation increases with increasing molybdenum oxide content throughout the temperature range studied.

Effect of the Structure of the Alumina Base and Peptization Thereof on the Properties of the Hydroforming Catalyst

In the preparation of alumina-molybdenum oxide hydroforming catalyst by impregnation methods, the alumina at the time of impregnation may have one of three forms: The trihydrate (Gibbsite, $Al_2O_3 \cdot 3H_2O$), the monohydrate (Boehmite, $Al_2O_3 \cdot H_2O$), or anhydrous (γ-Al_2O_3). To

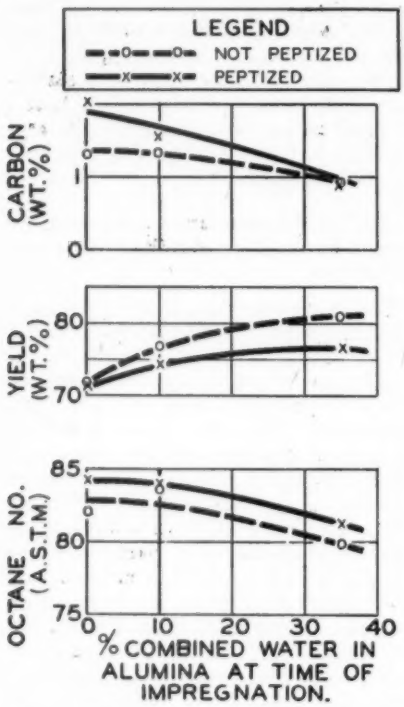


Fig. 2—Effect of structure of alumina base on octane number, yield, and carbon (impregnated catalysts)

TABLE 5—Effect of Structure and Peptization on Catalyst Activity

	γ-Al_2O_3		Boehmite		Gibbsite	
% H_2O when Impregnated	1.0	1.0	10	10	35	35
Peptization with HAc	No	Yes	No	Yes	No	Yes
Bulk Density, gm./ml.	0.99	0.97	1.01	0.95	1.10	1.03
Specific Surface, M^2/gm.	102	...	149	...	64	...
Mean Pore Diameter, A	159	...	105	...	197	...
Activity Data						
Wt. % Gasoline	80.9	76.4	76.8	74.6	71.8	71.4
Wt. % Carbon	0.92	0.90	1.34	1.55	1.32	2.02
O.N., F-2	79.9	81.3	83.7	83.5	81.6	84.1

determine whether the form of the alumina at time of impregnation, and whether treatment of the alumina with a peptizing agent, e.g., acetic acid, has any effect on the activity and stability, a systematic investigation was made.

Catalysts containing 9% MoO_3 were prepared by impregnating powdered alumina trihydrate, monohydrate, and anhydrous γ-Al_2O_3—both peptized (with acetic acid) and not peptized—with ammonium molybdate solution. The alumina trihydrate (Gibbsite) was a product of the commercial Bayer process. The alumina monohydrate (Boehmite) was prepared from the trihydrate by calcination at 600° F.; the anhydrous γ-Al_2O_3 was prepared from the trihydrate by calcination at 1300° F. The peptization of the various aluminas was accomplished by soaking the powdered material in a 50% glacial acetic acid solution for one hour and stirring frequently. At the end of this time the samples were filtered and washed twice with water.

After impregnation the catalyst powders were dried on a steam bath, mixed with lubricant and formed into ⅛ x ⅛-in. pills. The pelleted catalyst was freed from lubricant and partially dehydrated at 932° F. and finally calcined for 6 hours at 1112° F. The catalysts were then tested for activity as previously described.

The activity results are shown in Table 5 and Fig. 2, where the octane number of the hydroformed product, per cent yield, and carbon are plotted as a function of the combined water in the alumina at the time of impregnation. In every case the circles connected by broken lines and the crosses connected by solid lines refer to the unpeptized and peptized alumina respectively.

It is observed that all of the data are consistent in indicating that peptization of the alumina yields a catalyst of higher activity than does unpeptized alumina. Also, it is seen that the structure of the alumina, i.e., the percentage of combined water at the time of impregnation has an effect upon the activity, lower activity being associated with higher water content. It is difficult from these data alone to select the best catalyst. However, cross plots of octane number vs. yield, and octane number vs. carbon indicate a possible slight superiority for catalysts made from peptized alumina monohydrate.

Effect of Siliceous Diluent on Catalyst Activity and Thermal Stability

As mentioned earlier, the structural stability of the hydroforming catalyst may be improved by the addition of a small amount of clay (or other siliceous material). To determine whether the activity characteristics after high temperature calcination were also improved by the presence of clay, three experimental catalysts were studied in detail, both structurally and from an activity standpoint.

Three catalysts were prepared from alumina trihydrate by impregnation with ammonium molybdate solution. Catalyst No. 1, the control sample, contained no clay. Catalyst No. 2 contained 10% clay (Bentonite) dry basis, blended in after impregnation. Catalyst No. 3 contained 10% of dried silica gel powder. All three catalysts were dried at 300° F., mixed with lubricant and then formed into ⅛ x ⅛-in. pills.

The catalysts were then freed of lubricant and calcined for 6 hours at 1112°, 1292°, 1382°, and 1472° F. The molybdenum oxide content of catalysts 1, 2, and 3 calcined at 1112° F. were 9.6, 10.2 and 11.8% respectively.

All samples were examined by X-ray diffraction and for activity by the methods previously described. Because of the very low strength of the samples calcined at the higher temperatures, it was not possible to make the activity tests on the pelleted catalysts. Instead the calcined catalysts were ground, pressed in a hydraulic press and then broken into granules of 6 to 8 mesh.

The X-ray examination showed that all of the catalysts have the γ-Al_2O_3 structure after the 1112 and 1292° F. calcination. However, the catalysts containing no diluent nor dried silica gel show the presence of the previously described alumina-molybdena complex after the 1382°F. calcination, while the catalyst containing

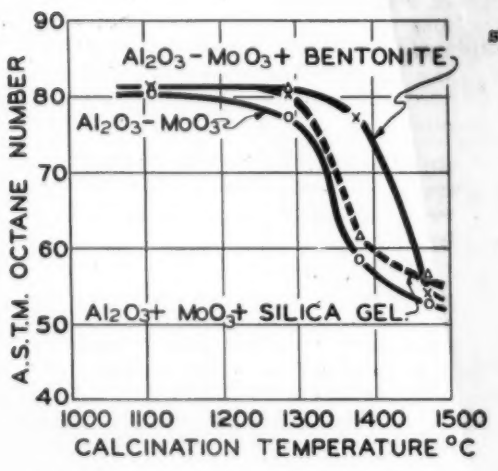


Fig. 3 (left)—Effect of calcination on octane number (various impregnated catalysts)

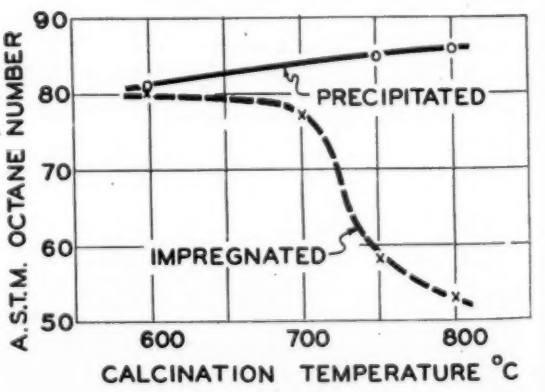


Fig. 4 (right)—Comparison of the thermal stability of impregnated and precipitated catalysts

Bentonite clay does not. All three catalysts contain the complex after calcination at 1472° F. It thus appears that Bentonite clay is capable of stabilizing the alumina-molybdenum oxide catalyst to the extent that it may be heated to a temperature about 80° F. higher than catalysts not so stabilized.

These conclusions are confirmed by the results of the activity tests shown in Fig. 3. Here it is seen that the catalyst which contains clay may be heated to a temperature about 80° F. higher than the unstabilized catalyst for the same decrease in the octane number of the product. The exact reason for this stabilizing effect is not known, and there may well be other materials capable of stabilizing the catalyst that are even superior to Bentonite clay.

PART II

PRECIPITATED-TYPE CATALYSTS

THE work described in the foregoing section led to the general conclusion that, although alumina-molybdenum oxide catalysts could be improved with respect to stability in certain ways, the extent of improvement was small. The whole problem was, therefore, reconsidered, and it was decided that really deep-seated changes in the catalyst would have to be made if the desired increase in stability were to be attained. The most significant feature of the preceding work is that catalyst stability can be improved by (1) improving the uniformity of distribution of the molybdena throughout the alumina and (2) decreasing the alumina crystallite sizes.

It was decided that the only way in which these objectives could be attained was to abandon impregnation techniques and use instead what has been called precipitation techniques. By this is meant that the alumina should be derived from an aluminum salt by precipitating the alumina by addition of a precipitant, such as ammonia. One may thereby have control at all times of the factors which influence crystallite size. This technique also permits incorporation of the activating compound at any stage in the preparative procedure, so that the uniformity of the mass may be assured.

The general procedure employed in making the precipitated-type catalyst was as follows: (8, 9)

Commercial aluminum chloride hexahydrate ($AlCl_3 \cdot 6H_2O$) was dissolved in water and then filtered to remove splinters of wood and other foreign matter usually present in this material. The desired amount of ammonium molybdate solution was poured into aluminum chloride solution. With thorough mixing, sufficient precipitant (NH_4OH or $(NH_4)_2CO_3$) was added over a period of about 5 minutes to reach the desired pH. The wet catalyst mass was dried at 250° F. for 12 to 18 hours and calcined at 700-750° F. for 8 hours to remove the volatile ammonium chloride.

The catalyst was ground to pass a 30-mesh sieve, mixed with lubricant and pilled into ⅛ x ⅛-in. cylindrical pellets. The pills were freed of lubricant and residual water by calcining them for 2 hours at 1112° F. in a stream of air. The catalysts were then either tested directly for activity or additionally heat-treated at temperatures up to 1472° F. and then tested for activity to determine their stability characteristics. Several variations were made in this preparative procedure; the effect of these variations on activity, area, X-ray structure, etc., is discussed below.

Thermal Stability—Preliminary Survey

To determine rapidly if this precipitation method of preparing catalysts had any special merit, one sample was made by the method described immediately above and calcined in air at 1112°, 1382°, and 1472° F. for 6 hours. The catalyst contained 7.3% MoO_3, the remainder being Al_2O_3. The calcined catalysts were then tested for activity by the same method used in the work on impregnated catalysts. The results are shown in Table 6.

These data are plotted in Fig. 4 along with typical data on an impregnated catalyst. The tremendous improvement in stability is striking. It should be noted that the new type catalyst is appreciably improved as the calcination temperature is raised. As a result of these encouraging data, extensive tests were made to study the properties of this new catalyst in detail. The more interesting items are discussed below.

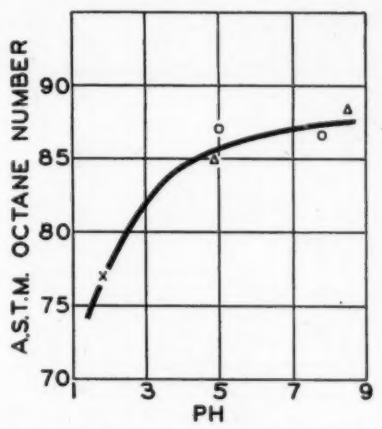


Fig. 5—Effect of pH of precipitation on octane number (precipitated catalyst)

Effects of pH and Precipitant

Among the first points to be investigated were the effects of type and amount of precipitant used in coagulating the alumina. Both ammonium hydroxide and ammonium carbonate were used for this purpose. Also, one test was made in which no precipitant at all was used. The quantity of the precipitating agent was varied to cause the final pH to range from about 2 to about 8. The actual tests made, together with the activity and other inspection data, are given in Table 7. The variation of ASTM octane number (F-2) with pH is shown in Fig. 5. It is interesting to note that the octane number rises fairly rapidly with increasing pH. The specific surface also increases with the pH, and at first glance it might be concluded that the catalysts made at low pH are of low activity because their specific surface is small. X-ray diffraction data, however, suggest a different explanation.

The X-ray studies show that the catalyst made at the lowest pH contains anhydrous γ-alumina and other

TABLE 6—Activity Data on Calcined Catalysts

Catalyst No. 959	Temp. of Calcination ° F.	ABD Gms/ml.	Crushing Strength (lbs.)	Activity Data		
				Gasoline Vol. %	Carbon Wt. %	O.N. ASTM
120 A	1112	0.86	9.9	75.7	...	81.2
120 B	1382	0.88	6.8	67.8	0.72	84.7
120 C	1472	0.90	5.9	69.4	1.24	85.9

TABLE 7—Effects of pH and Precipitant on Catalyst Activity

Catalyst No. 959	Precipitant	pH	Calcination Temp. ° F.	ABD Gms/ml.	Activity Data			Surface Data	
					Gasoline Vol. %	Carbon Wt. % of Charge	O.N. (10# RVP)	Aren Sq. In./Gm.	Mean Pore Diameter A
110	None	1.8	1382	0.75	82.0	0.30	76.9	36	597
116	NH_4OH	4.9	1382	0.91	71.4	0.88	84.5	123	134
122	NH_4OH	8.5	1382	0.81	63.6	0.94	88.3	151	122
128	$(NH_4)_2CO_3$	5.0	1382	0.91	69.6	1.33	87.0	...	...
134	$(NH_4)_2CO_3$	7.8	1382	0.54	68.0	0.71	86.6	183	175

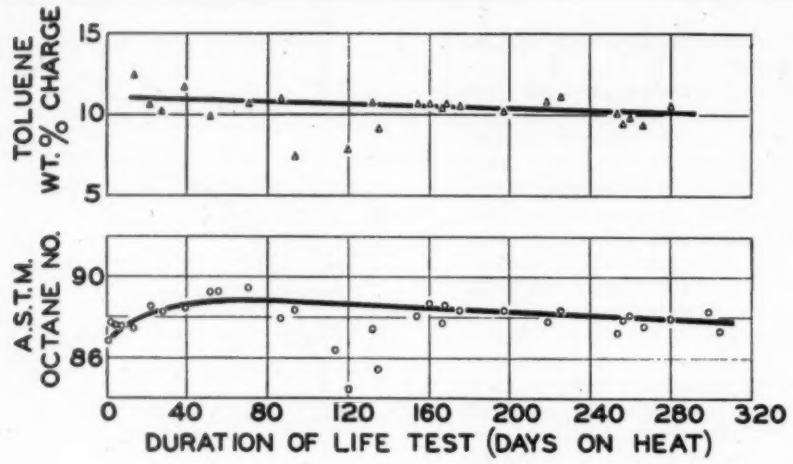


Fig. 6—Life test on precipitated hydroforming catalyst

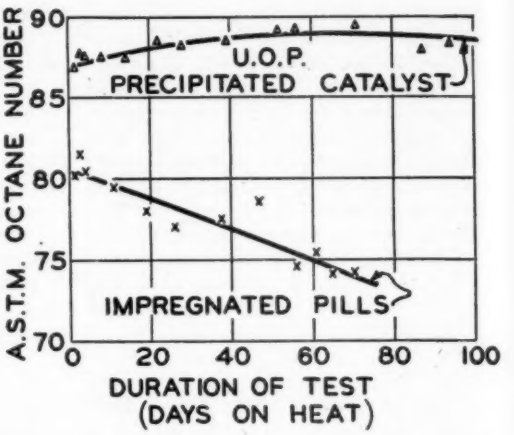


Fig. 7—Comparative life tests on precipitated and impregnated catalysts

unidentified materials, presumably oxychlorides, after drying at 250° F. The catalysts made at the higher pH values contain only the monohydrate (α-$Al_2O_3 \cdot H_2O$), or Boehmite after a similar drying treatment. After calcination at 1382° F., all the catalysts show the same X-ray structure, corresponding to γ-Al_2O_3. Thus it would appear that catalysts formed at low pH values are not homogeneous mixtures of the support (alumina) and promoter (molybdenum oxide) but also contain compounds such as aluminum oxychlorides.

The X-ray tests show that all of these highly active catalysts made by precipitation techniques contain γ-Al_2O_3 as the only identifiable crystalline phase. This is the same result that was obtained with the less active catalysts made by impregnation procedures. The X-ray data do show one important difference between the two types of catalysts, namely, the markedly smaller crystallite size of the γ-Al_2O_3 in the more active materials.

As is well known, the breadth of the diffraction lines appearing on an X-ray diffraction film depends on the size of the diffracting crystallites. Narrow sharply defined lines are obtained from materials containing large crystallites while broad diffuse lines appear if the crystallites are very small. Study of the widths of the lines obtained with the two types of catalysts revealed that the crystallite size of the γ-Al_2O_3 was 5 to 10 times smaller for the precipitated type than for the impregnated type.

Some attempts were made to examine these catalytic materials with our Type B RCA electron microscope. It was believed that the distribution of the molybdenum oxide on the alumina might be quite dependent on the manner in which the two components are mixed. In the impregnation process, the molybdenum compound is adsorbed onto a preformed surface. This method undoubtedly yields a non-uniform composite in the sense that the coverage of the alumina surface by the activating compound is spotty. Such a condition does not exist for the precipitated type composites, since in this case the activator and support are intimately mixed while each is in a highly dispersed state.

It seems reasonable to suppose, then, that molybdenum oxide may exist on impregnated alumina supports in the form of globules that are very much larger than those in the precipitated type catalyst. It was the purpose of the electron microscope tests to learn whether such globules could be observed. The results were all negative. While this does not mean that large clusters of molybdenum oxide do not exist, it does show that such aggregates, if present at all, are smaller than about 40 Angstrom units, which is the limit of resolution of our microscope at the time these tests were made.

TABLE 8—Life Test No. 1 Data Summary, New UOP Hydroforming Catalyst

Period No.	Days on Stream	Liquid Yield Wt. % of Chg.	Toluene Yield Wt. % of Chg.	Carbon Wt. % of Chg.	ASTM O.N.
1-2	1	71.6	....	0.79	86.8
3-4	3	67.1	....	0.82	87.7
5-6	4	69.0	....	1.19	87.5
18-19	8	68.3	....	0.88	87.5
34-35	14	66.3	12.35	0.83	87.4
53-54	22	65.8	10.57	0.87	88.5
71-72	28	66.5	10.21	0.92	88.2
98-99	39	66.2	11.65	0.93	88.4
134-135	52	66.7	9.82	1.06	89.2
145	56	68.0	....	0.96	89.2
177-178	71	66.7	10.67	0.84	89.4
220-221	87	68.9	10.86	0.79	87.9
236-237	94	66.6	7.36	0.75	88.3
287-288	114	78.3	9.40	0.82	86.4
303-304	121	74.0	7.80	1.24	84.5
328-329	133	77.4	10.73	0.86	87.4
333-334	135	75.2	9.11	1.41	85.4
379-380	155	69.9	10.63	0.97	88.0
394-395	161	69.9	10.53	0.87	88.6
406-407	167	67.3	10.30	1.00	87.7
411-412	169	67.7	10.56	0.96	88.6
428-429	176	70.4	10.40	0.94	88.3
480-481	198	67.9	10.17	0.87	88.3
529-530	219	68.8	10.73	1.00	87.7
544-545	226	69.2	10.96	0.93	88.3
610-611	254	70.0	10.00	1.00	87.2
621-622	257	69.9	9.31	0.92	87.8
625-626	260	68.2	9.72	1.00	88.0
639-640	267	70.0	9.15	...	87.5
672-683	281	67.4	10.45	1.01	87.9
715	299	69.0	....	1.15	88.2
725	305	69.6	....	1.04	87.3

TABLE 9—Life Test No. 2 on Impregnated Al_2O_3 Pills Containing 9.5% MoO_3

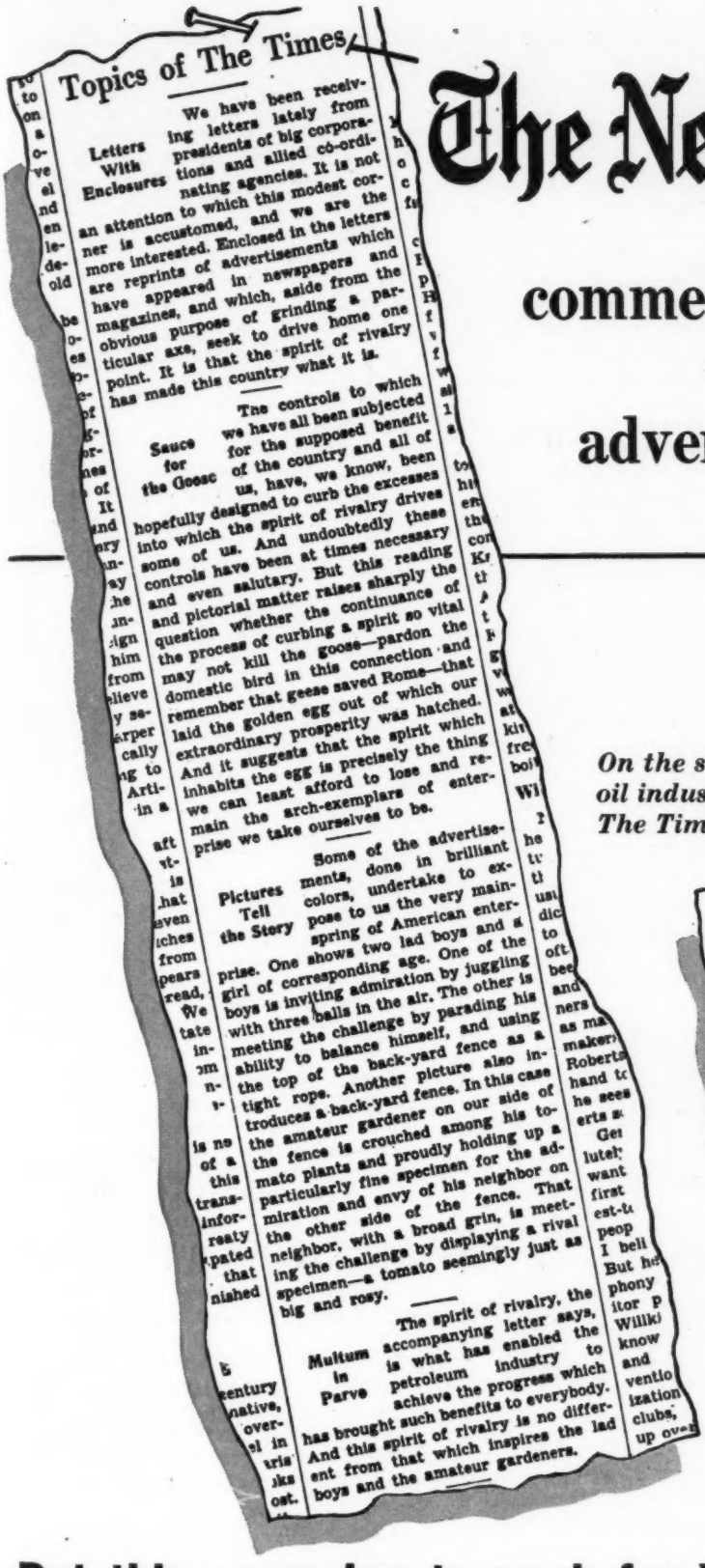
Period No.	Days	Liquid Yield Wt. % of Chg.	Carbon Wt. % of Chg.	ASTM O.N.
2-4	1.5	79.0	1.56	80.5
5-7	2.6	76.3	...	81.5
8-10	3.7	77.5	...	80.5
25-30	11.0	78.2	1.75	79.5
49-51	19	78.7	2.04	78.0
69-71	26	80.7	1.87	77.0
99-101	37	76.6	1.13	77.5
124-126	47	80.8	0.94	78.7
149-151	56	81.0	1.20	74.7
163-165	61	83.2	1.33	75.5
174-176	65	83.2	1.29	74.1
187-189	70	81.0	1.32	74.2

Life Tests

The final decision as to whether a catalyst is an improvement over

The New York Times

comments on Petroleum's new advertising campaign



Topics of The Times

Letters With Enclosures

We have been receiving letters lately from presidents of big corporations and allied co-ordinating agencies. It is not an attention to which this modest corner is accustomed, and we are the more interested. Enclosed in the letters are reprints of advertisements which have appeared in newspapers and magazines, and which, aside from the obvious purpose of grinding a particular axe, seek to drive home one point. It is that the spirit of rivalry has made this country what it is.

Sauce for the Goose

The controls to which we have all been subjected for the supposed benefit of the country and all of us, have, we know, been hopefully designed to curb the excesses into which the spirit of rivalry drives some of us. And undoubtedly these controls have been at times necessary and even salutary. But this reading and pictorial matter raises sharply the question whether the continuance of the process of curbing a spirit so vital may not kill the goose—pardon the domestic bird in this connection—and remember that geese saved Rome—that laid the golden egg out of which our extraordinary prosperity was hatched. And it suggests that the spirit which inhabits the egg is precisely the thing we can least afford to lose and remain the arch-exemplars of enterprise we take ourselves to be.

Pictures Tell the Story

Some of the advertisements, done in brilliant colors, undertake to expose to us the very mainspring of American enterprise. One shows two lad boys and a girl of corresponding age. One of the boys is inviting admiration by juggling with three balls in the air. The other is meeting the challenge by parading his ability to balance himself, and using the top of the back-yard fence as a tight rope. Another picture also introduces a back-yard fence. In this case the amateur gardener on our side of the fence is crouched among his tomato plants and proudly holding up a particularly fine specimen for the admiration and envy of his neighbor on the other side of the fence. That neighbor, with a broad grin, is meeting the challenge by displaying a rival specimen—a tomato seemingly just as big and rosy.

Multum in Parvo

The spirit of rivalry, the accompanying letter says, is what has enabled the petroleum industry to achieve the progress which has brought such benefits to everybody. And this spirit of rivalry is no different from that which inspires the lad boys and the amateur gardeners.

On the subject of the oil industry's advertising, The Times continues . . .

Mark Twain Is Responsible

No mention is made by the petroleum spokesman of the use to which Tom Sawyer put the spirit of rivalry when it came to a whitewashing job. That, we submit, is a classic example of American enterprise, and one which, in the past, has not lacked admiration—or imitation in circles less juvenile. In such cases it has been roundly denounced as exploitation.

In Tom Sawyer, too, the American spirit spoke—and spoke in native accents. But the accent today is no longer on exploitation. Rivalry has become not a race for private profit but the firm foundation upon which rests the entire fabric of our national economy. It must be preserved.

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Just Mail the Coupon

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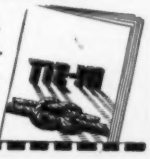
Title .....
Company .....
Address .....
City ..... *State* .....



other existing preparations must be determined by its actual performance in process operation. This test was conducted with the UOP catalyst in a life test lasting 305 days on heat and 181 days on the process cycle. The catalyst chosen for this test was one which did not show phenomenally high activity but was rather an average catalyst that could be reproduced without difficulty.

In order that the test might have further significance, the catalyst chosen was a part of a large batch from which portions were submitted to several oil companies for testing. The apparatus used for the test was designed to operate automatically through a cycle comprising the process, purge, and regeneration periods. The details of the test procedure are as described for Life Test No. 1. The operation of the unit was quite satisfactory and no known periods of misoperation occurred.

The efficiency of the catalyst was measured by two properties of the hydroformate recovered from the unit; i.e., first, the octane improvement and second, the toluene production. The first property is of special interest to the peacetime utilization of the process, the production of motor fuel, while the second was pertinent to the production of toluene during the war. The results of the life test are summarized in Table 8 and Fig. 6. The data show that the octane number of the hydroformate increased from an initial of 87 to 89 during the first 50 days operation and thereafter dropped at the rate of about one octane number per 6 months operation. The toluene production shows considerable variation but during the 305 days the production of toluene fell off about 1% by weight of the charge.

Similar life test data have been obtained on a variety of impregnated type catalysts, except that the duration of these tests was much shorter than that on the precipitated catalyst. For purposes of illustration, data obtained on a typical impregnated catalyst are given in Table 9. These data are plotted in Fig. 7, in which is also included a portion of the life test results obtained on the precipitated catalyst. It is seen that the impregnated catalyst loses activity at a far greater rate than the improved one.

SUMMARY

This paper summarizes work done during the past several years on the development of highly active and stable alumina-molybdenum oxide hydroforming catalysts which are useful in the manufacture of aromatics and motor fuels.

Conventional catalysts, made by impregnating alumina granules or pellets with a molybdenum compound followed by drying and calcining are deficient in several respects, notably in their low activity, high coke-forming properties, and poor catalytic and strength stability.

The improved catalysts, made by precipitating alumina hydrogel in the presence of ammonium molybdate, followed by drying, calcining, and pelleting, are characterized by having very high activity, low coke-forming properties, and excellent stability. A life test, involving continuous use of this type catalyst, was conducted for a period of 305 days. The F-2 octane number of the hydroformate was 86.8 for fresh catalyst and 87.3 for catalyst used 305 days. A comparable test on an impregnated type catalyst, showed that the F-2 octane number of the hydroformate dropped from an initial value of 80.5 to 74.2 in 70 days.

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MEET THE AUTHORS

Dr. Glenn M. Webb, director of the Physics Division of Universal Oil Products Co., graduated from California Institute of Technology in 1931 with B.S. degrees in physics and mathematics. Postgraduate work in the same subjects at Washington University, St. Louis, earned him the M.S. degree in 1933 and the Ph.D. degree in 1935.

Dr. Webb

From 1935-37 he was research physicist at Western Cartridge Co., East Alton, Ill., joining UOP in 1937. For a year and a half during the war Dr. Webb, in addition to his work at Universal, was engaged as a consultant on catalysts in the Manhattan atomic bomb project.

Dr. C. H. Ehrhardt, research physicist with Universal Oil Products Co., graduated in 1933 from Hillsdale College, Hillsdale, Mich., with a bachelor degree in physics. The next 5 years he spent at Purdue University as a graduate assistant in physics, earning his Ph.D. degree in 1939.

Dr. Ehrhardt

Mr. Smith

In 1940, after a few months as instructor in physics at Purdue, he joined UOP. His special fields of research work there are X-ray diffraction, electron diffraction and electron microscopy.

Marvin A. Smith graduated from Wabash College in 1929 with an A.B. in chemistry, and joined the research staff of Universal Oil Products Co. the same year. His first work was in distillation, desulfurization and treating of motor fuels. His other assignments have included the operation of pilot plants in thermal cracking, polymerization and other processes, and investigation of Diesel fuels and Diesel fuel dopes.

Since the Physics Division of the UOP laboratories was established in 1939, Mr. Smith has been a member of that division, engaged in the development and study of catalysts.

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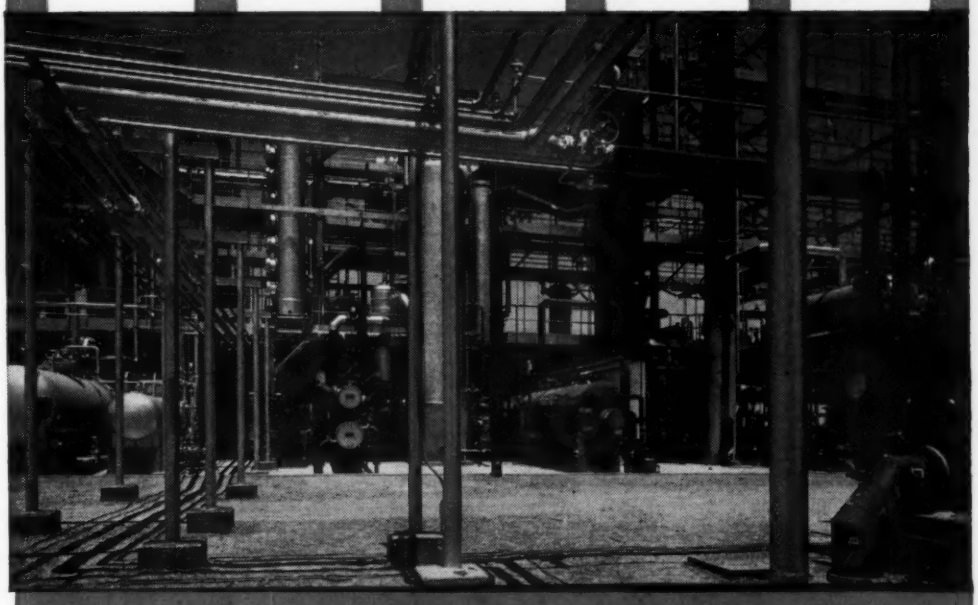

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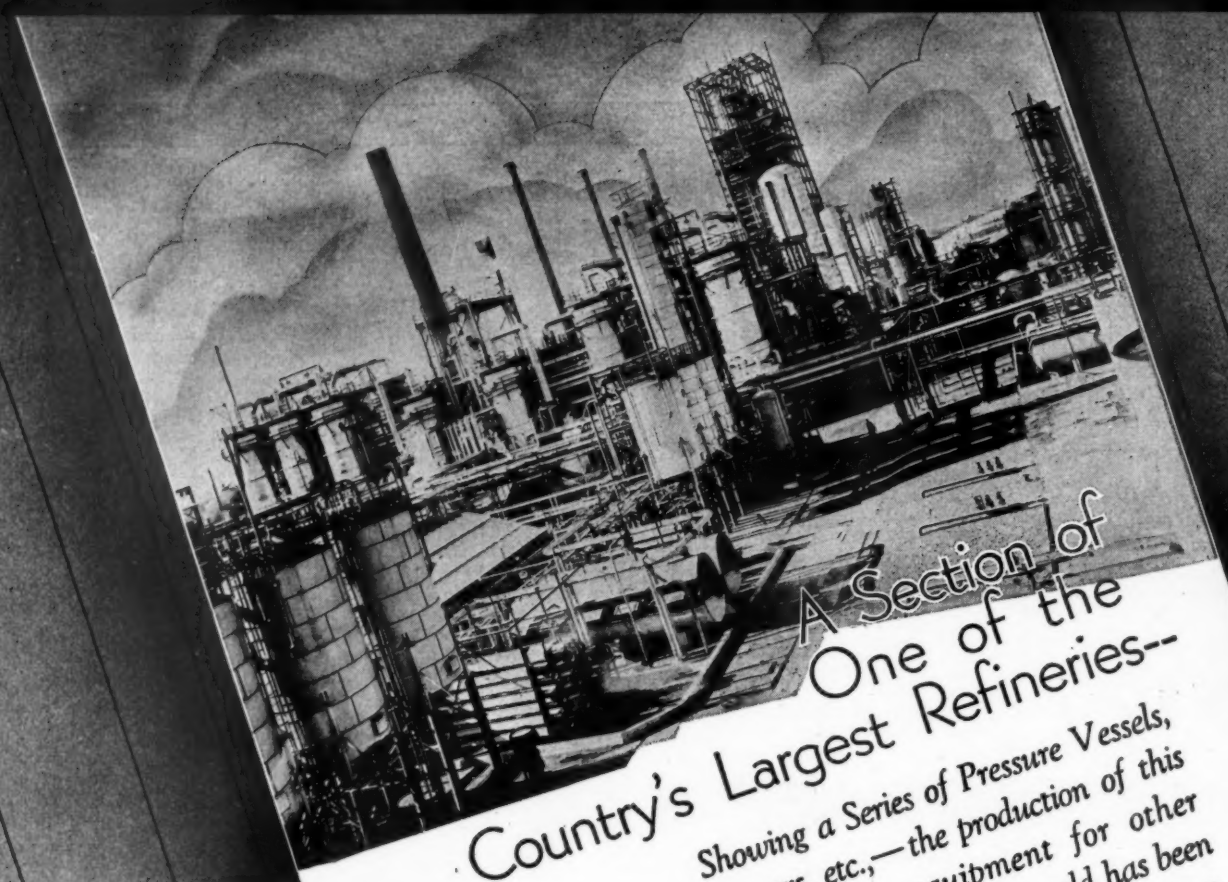

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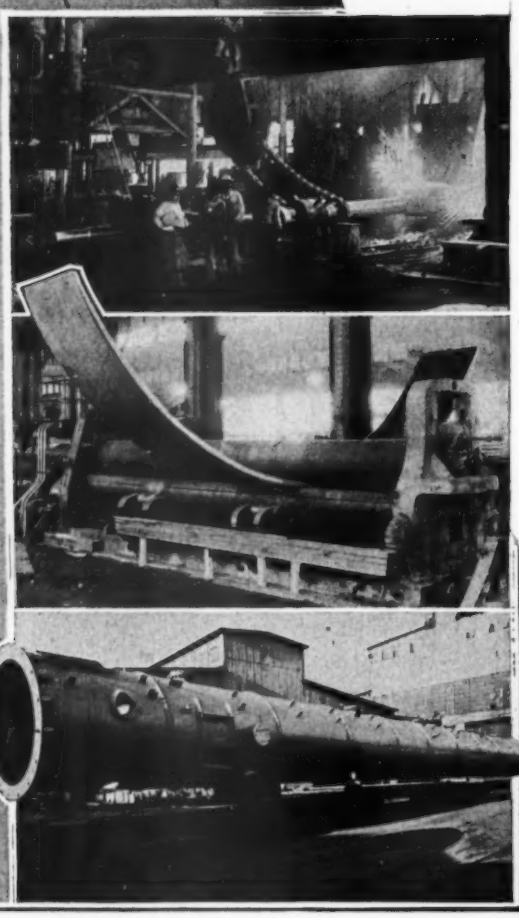

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CERTIFIED ISO-OCTANE by the CAR LOAD

Article 2—Handling and Transportation, Storage and Dispensing, Composition

By OTTO GERBES, H. J. HALL, and A. E. BECKER

Very great precautions are taken at all steps in handling and storing certified iso-octane destined for use as primary reference fuel, to insure maintaining the high degree of purity achieved in its manufacture. Freezing point determinations, taken before shipment and on arrival at point of storage, must agree, to show no contamination has occurred during shipment. Equally elaborate measures are taken to preclude contamination in storage and dispensing.

Analyses of the composition of one batch of certified iso-octane, made by chemical, infrared and freezing point methods, determined that the effect of impurities present on octane rating was negligible. These analyses indicated total impurities amounted to 0.38 mol%. Purity may be improved to 99.9%, where required, by a distillation and filtering procedure. Article 1, in the October issue of *Petroleum Processing* (pp. 734-736), described the commercial production of certified iso-octane.

AFTER manufacture, the iso-octane product is alternately received in two 10 x 30-ft. drums which serve as rundown tanks. After a drum is full and has passed the infra-red test, it is pumped directly into a thoroughly cleaned tank car. Freezing point apparatus was not available at Baytown at the time the current batch, No. 2, of certified iso-octane was manufactured. Therefore samples from each tank car were sent to the Baton Rouge laboratory of the Standard Oil Co. of New Jersey. Cars were sealed and released for shipment only after this laboratory reported that the samples met the ASTM freezing point specification, —107.52° C. minimum.

On arrival at the reference fuel storage plant of purchaser at Bayway, N. J., samples were again withdrawn from each car for freezing point determinations at the Standard Inspection laboratories at Bayonne. When the freezing point confirmed that no contamination had occurred during transit the contents of the car were pumped into the storage tank. The agreement between the two laboratories for the tank cars used in preparing Batch No. 2 is shown in Table 6.

Production and shipping of high purity products require many precautions. It is of interest to note that 3 to 4 days of operation are required to bring the iso-octane purity above 99% in the fractionation system when it is being transferred from some other service such as isopentane splitting to iso-octane service. When the system has been in iso-octane service but is coming on stream after a shut down, two days of operation are required to bring the purity up to standard. This is not surprising after consideration is given to the large size of the towers, 8½ ft. to 9½ ft. diameter, relative to the small production rate, 163 b/d.

All transfer lines, pumps, and equipment were initially, steamed, dried and then purged with iso-octane until the product purity remained high. Wherever possible, empty tank cars in iso-octane service are kept under seal for the return trip to the point of production where they can be reused. This saves many hours of cleaning. The technique for cleaning the tank cars has been proved by four years of experience in cleaning cars to be used for shipping high purity nitration grade toluene. The tank cars designated for iso-octane service have previously been in gasoline service. The overall time consumed in cleaning the car is about 20 manhours. The cleaning system is briefly as follows:

(1) The car is opened and two nozzles are lowered through the manhole. The steam nozzle is positioned at the bottom of the car and the water nozzle is positioned in the upper part of the car.

(2) The car is steamed for three hours.

(3) After steaming, the car is cooled with water requiring approximately 30 minutes.

(4) After a safe gas test is obtained, a man enters the car to wash the entire inner surface using high pressure water for 30 minutes. This eliminates scale and provides a general washdown.

(5) The car is again steamed until hot at which time the car is filled with cold water. Steam is continued until the water boils, after which the hot water is drained. This steaming and draining operation requires five hours.

(6) Step 5 is repeated two more times, starting with the cold water fill, followed by boiling and draining. The action of the cold water on the hot car followed by boiling is considered important in cleansing the seams and rivets.

(7) After the last hot water drain, the car is water cooled and re-entered for a high pressure water wash.

(8) The car is allowed to drain after which it is entered for a "soak up" of water pools using clean, dry rags.

(9) The car is finally warmed with steam to approximately 140°F. drained and air dried.

Storage and Dispensing

Many precautions are also required for the storage and dispensing of high purity products. Storage tanks, lines to and from them, dispensing equipment and shipping containers must be thoroughly cleaned prior to use. There should be no possibility of contamination of one product with another or with other re-

TABLE 6*—Freezing Points, °C, of Tank Car Shipments

Car	Before Shipment	On Arrival
1	—107.501	—107.483
2	.492	.501
3	.510	.511
4	.473	.471
5	.467	.467
6	.467	.454
7	.474	.491
8	.443	.470
9	.431	.429
10	.425	.452
11	.425	.448
12	.441	.437
13	.447	.427
14	.454	.490
15	.442	.431
16	.485	.443
17	.496	.481
18	.475	.468
19	.480	.437
20	.464	.461
21	.460	.456
22	.478	.467
23	.479	.474
24	.498	.491
25	.493	.483
26	.475	.484
27	.476	.499
28	.451	.457
29	.465	.475
Average	—107.468	—107.466

* Tables 1 through 5 appeared with the first "Certified Iso-octane by the Car Load", article, in October PETROLEUM PROCESSING, pp. 734-736.

finery products, and change of composition during storage must be avoided.

Such precautions have been in use for many years in the handling and dispensing of secondary reference fuels. Early in 1930, it was necessary to prepare new batches, A-2 and B-2, of the Standard Reference Fuels then in use in the laboratories of the Standard Oil Co. (NJ) and its affiliates. Four pressure storage tanks of 18,000 gallons capacity each were provided. This was done to insure a supply that would be adequate for the laboratories of the Standard Oil Co., (N. J.), affiliates for several years, since the octane scale had not yet been adopted. Storage under pressure was used to avoid breathing, thereby minimizing change in composition due to loss of light ends. Reference Fuels A and B were prepared from virgin naphthas, as has been the case for succeeding batches of A, C, and M, thereby reducing the possibility of oxidation and change in composition.

Early in 1931, Standard Reference Fuels A-2 and B-2 were made available to the members of the Detonation Sub-Committee of the CFR Committee for conducting cooperative tests connected with the development of a suitable standardized Knock Test Method for motor fuels. The use of reference fuels that would be available over an extended period had obvious advantages. It enabled the Committee to evaluate the reproducibility of the methods that were being investigated. It also resulted in many laboratories throughout the industry using these fuels in conducting their own tests, thereby reducing much of the confusion inherent in comparing ratings obtained with different reference fuels.

TABLE 7—Inspection data for Certified Iso-octane

	Batch No. 1		*Purified Iso-octane
	Std. Oil Dev. Co.	Nat. Bur. of Stds.	Nat. Bur. of Stds.
ASTM Motor octane number	100.0	100.0	.....
Density at 20C, g/ml.	0.69186	0.69184	0.69182
Freezing point, C.	−107.473	−107.468	−107.311
Av. of tank cars on shipment	−107.483	.....	.....
Av. of tank cars on arrival	−107.482	.....	.....
Boiling pt. at 760 mm. Hg., C.	.....	99.222	99,234
Temp. increase, 20 to 80% recovery, C.	.....	0.005	.....
Refractive index at 20C.	.....	1.39141	1.39146
Aniline point, C.	80.3	80.0	.....

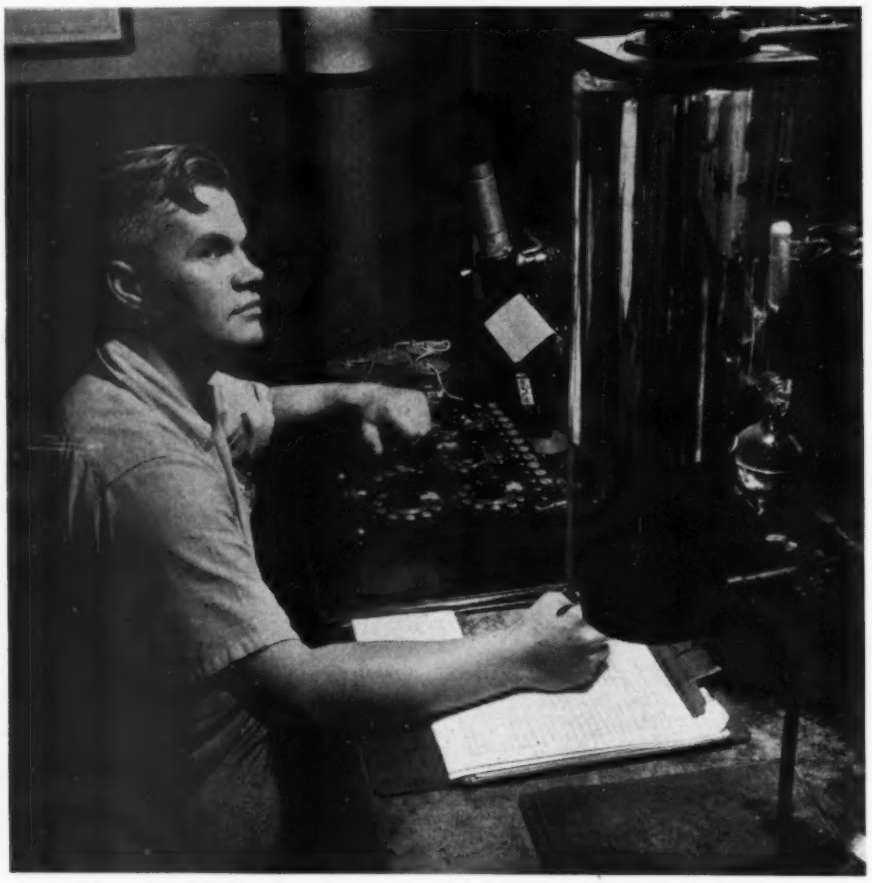
	Batch No. 2		
	Std. Oil Dev. Co.	Nat. Bur. of Stds.	ASTM Specs.
ASTM Motor octane number	100.0	100.0	100.0 ± 0.1
Density at 20 C, g/ml.	0.69191	0.69183	0.69190 ± 0.00010
Freezing point, C.	−107.465	−107.459	−107.52 min.
Av. of tank cars on shipment	−107.468	.....	.....
Av. of tank cars on arrival	−107.466	.....	.....
Boiling pt. at 760 min. Hg., C.	.....	99.241	99.25 ± 0.05
Temp. increase. 20 to 80% recovery, C.	.....	0.013	0.06 max.
Refractive index at 20 C.	1.39141	1.39156	1.39150 ± 0.00010
Aniline point, C.	79.9	.....	.....

* From "Properties of Purified Normal Heptane and Iso-octane (2,2,4-Trimethylpentane)" by D. B. Brooks, J. of Research, Nat. Bur. of Stds., Vol. 21, p. 850, Dec., 1938.

The adoption of the octane number scale in 1931 and the subsequent calibration of A-2 and B-2 by the Committee's method accelerated the use of these fuels. Also Standard Reference Fuel C was made available to meet the need for a secondary reference fuel of higher octane number than B-2. To provide for the increased demand and to take more adequate precautionary measures, the present storage and dispensing facilities were built in 1936. The plant is a separate unit without any connecting lines to other refinery tankage or equipment. All products are received by tank car or tank wagon. The plant consists of a spheroid and two spherical tanks of large capacity, several smaller upright cylindrical tanks, and a filling house.

The large tanks were new and all of the cylindrical tanks were reconditioned by sand blasting, followed by cleaning methods similar to those previously described for cleaning tank cars. Each tank is an individual unit equipped with its own pump, filling and dispensing lines. Connecting lines permit the pumping of a product from one tank to another or the use of two tanks as a unit whenever occasion requires. However, all connecting lines are blank flanged except during use for these purposes. Drain cocks are placed at all low points.

The return line from each pump enters its tank above the center line and is equipped with a nozzle so directed that circulation of the tank contents results in thorough mixing. After tests of top, middle and bottom samples show that uniformity has been attained, the line from the pump to the dispensing nozzle in the filling house is washed thoroughly with the product. Any samples required for calibration purposes or



Inspector determining purity of certified iso-octane by freezing point method with the aid of a G-1 Mueller resistance bridge and platinum resistance thermometer

other laboratory testing are then drawn by the normal dispensing and shipping procedure. The product is filled directly into new 50-gal. or 5-gal. drums which are lacquer lined as an added precaution during shipment and use.

The lining was developed during the handling of secondary reference fuels to guard against any possible effect of metal on these products. This also permits the introduction of tetraethyl lead directly into the drum if the user has occasion to make a leaded reference fuel blend. To assure accuracy of measurement, separate scales are used for the 5-gal. and 50-gal. drums. The empty drum is placed on the scales and its tare weight determined. It is then filled to the required equivalent weight, sealed and stenciled immediately.

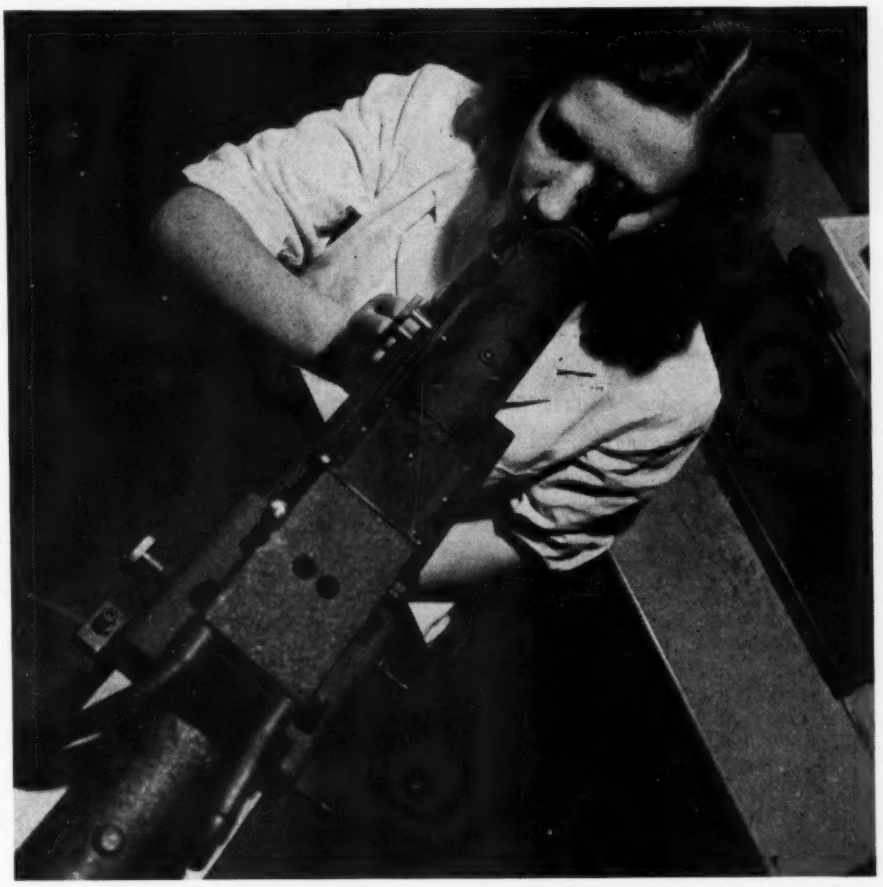
Two of the large tanks which previously contained secondary reference fuels F-6 and C-13 were used respectively for the storage of Batches Nos. 1 and 2 of certified iso-octane. Since F-6 was iso-octane of 97.4 mol % purity, no special cleaning operations other than completely draining and drying the tank were required. The tank used for storing Batch No. 2 had contained C-13. The following cleaning procedure was therefore used:

1. After completely draining the tank and lines, they were steamed for three days.

2. The entire tank was wire brushed, swept, wiped with rags, and inspected to make sure that all scale had been removed from the entire surface.

When the first tank car of certified iso-octane was received at Bayway in August, 1946, all lines to and from both storage tanks were flushed with the product until the freezing point of samples taken at the storage tank was the same as that of the material in the tank car. This flushing material, about 10 bbls., was then drained and the balance of the car pumped into the tank. As other cars arrived the contents were pumped into the storage tanks after making certain they met the ASTM freezing point specification.

Each of the two batches was circulated to insure uniformity. Samples were then drawn for testing at the National Bureau of Standards and at the Standard Inspection laboratories. The data obtained are compared in Table 7 with ASTM specifications and with similar data for a sample of high purity iso-octane prepared by D. B. Brooks of the National Bureau of Standards in 1938. It should be kept in mind that the freezing points of this table were determined from freezing curves, as distinguished from the melting point technique used in the subsequent portion of this paper.



Viewing samples of certified iso-octane through a precision interferometer. The instrument is used to aid in establishing purity of petroleum hydrocarbons

Composition of Certified Iso-octane

A detailed study of the composition of certified iso-octane Batch No. 2, has been made to determine the effect of any impurities present on octane ratings. The chief impurities are water, 2, 3-dimethylpentane, diisobutylene and toluene. The amounts of these and their probable effects on ASTM octane number are shown in Table 8.

The scheme of analysis used to obtain these data is indicated in Table 9. The sample was first distilled at high reflux ratio in a 90-plate packed column, taking small cuts at the front end and minimum bottoms to concentrate the impurities. The low boiling cuts, amounting to 3.92% of the sample, were analyzed by infrared; the only impurity found in significant amount was 2,3-dimethylpentane. Water amounting to 0.006 volume %, or 0.04 mol % on the original sample, was concentrated in the front ends as shown by chemical analysis. This would not show in estimates of purity by infrared or freezing point analyses.

The bottoms sample showed a trace of toluene by infrared, amounting to 0.06% on the bottoms. This is equivalent to 25 parts per million on the original sample and was confirmed independently by Raman analysis. There is no evidence of any paraffinic unknown in the bottoms; the chief impurity is apparently diisobutylene, The infrared shows a fairly strong peak for the major isomer of diisobutylene (2,4,4-trimethylpentene-1) and a smaller peak for the minor one (2,4,4,-trimethylpentene-2). A bromine number of 1.5 cg./ml. confirms the presence of olefinic impurity in the bottoms sample.

Measurements of differential refractive index against heart cut no.

TABLE 8—Effect on Octane Ratings of Impurities in Certified Iso-Octane

Impurity	Mol % on Sample	Est. ASTM Oct. No.*	Method of Analysis
Water (0.006 wt. %)	0.04	no effect	K. Fisher Reagent
2,3-Dimethylpentane	0.09	90	Infrared
Unidentified in light ends	0.04	unknown	Infrared, Fr. Pt.
Diisobutylene	0.13	<151, >89	Infrared, Br. No., Fr. Pt.
Toluene	0.003	<112, >103	Infrared, Raman
Impurities in heart cut	0.12	no effect	Fr. Pt.
Total hydrocarbons	0.383		

* Exact blending values in iso-octane at the 100-octane number level are not known.

24 as a standard were taken to the sixth place (precision of ± 0.000004) with a Rayleigh interferometer and used to pilot the analysis. These data indicate that the cuts from 3% to 96% were essentially of the same composition, with the fractions from 4% to 93% showing no detectable difference. The low refractive index of the first cuts is attributable to the dissolved water concentrated in them.

To determine the total impurity in the front ends and check the infrared analysis, a second distillation was made under identical conditions. The initial 3.92% cut was used for a freezing point measurement. Also, freezing points were taken on the next 5.8% cut, the two heart cuts, and the bottoms of the first distillation. All freezing points were measured from melting curves obtained under dry nitrogen, following essentially the method and procedure recommended to Sub-committee XXV of ASTM Committee D-2 by the National Bureau of Standards, rather than the freezing curves used for manufacturing control.

The conversion of freezing point to mol % impurity involves the use of some theoretical value previously determined for the freezing point of the pure compound. The data obtained in the course of the present study indicate that the true freezing point of 2,2,4-trimethylpentane may be somewhat higher than the −107.365° C.. suggested by F. D. Rossini, but not as high as the −107.311 reported by D. B. Brooks. The recommended procedure calls for using the melting curve in all cases where the purity of sample exceeds 99.5 mol %. The use of a dry nitrogen atmosphere instead of air over the sample is a safety precaution to avoid any chance of condensing liquid oxygen or other high boiling gases into the hydro-carbon when using liquid nitrogen as the coolant. This also raises the observed freezing point about 0.021°C. The interferometer and freezing point methods used in this analysis are being discussed in detail in a separate article (to be published in Industrial and Engineering Chemistry).

For the purpose of this study the true freezing point of iso-octane as determined from melting curves under dry nitrogen is taken as −107.344° C. Thus, the original sample at −107.438° C. calculates to 0.38 mol % impurity or 99.62 mol % purity. Total hydrocarbon impurities determined from freezing points on the individual cuts amount to 0.383%, Table 8. A further check is obtained by translating the results of infrared and other analyses into theoretical increments of refractive index. The increment calculated for the amounts of water, 2,3-dimethylpentane and trace of 3-methylhexane determined by analysis in the first 3.9% distillate is −0.000054. This compares with an observed composite value of −0.000057 for the same fractions. In the bottoms the increment in refractive index calculated for the toluene and diisobutylene identified is +0.00068, compared to +0.00066 observed. These checks are well within the accuracy of the measurements.

Stated differently, if the purities calculated here from freezing points are too high, the additional impurity must be one which is not separated to any detectable amount by a 90-plate distillation using 700:1 reflux ratio for the light ends and 100:1 reflux for the remainder. This excludes the paraffins, naphthenes and aromatics other than those indentified.

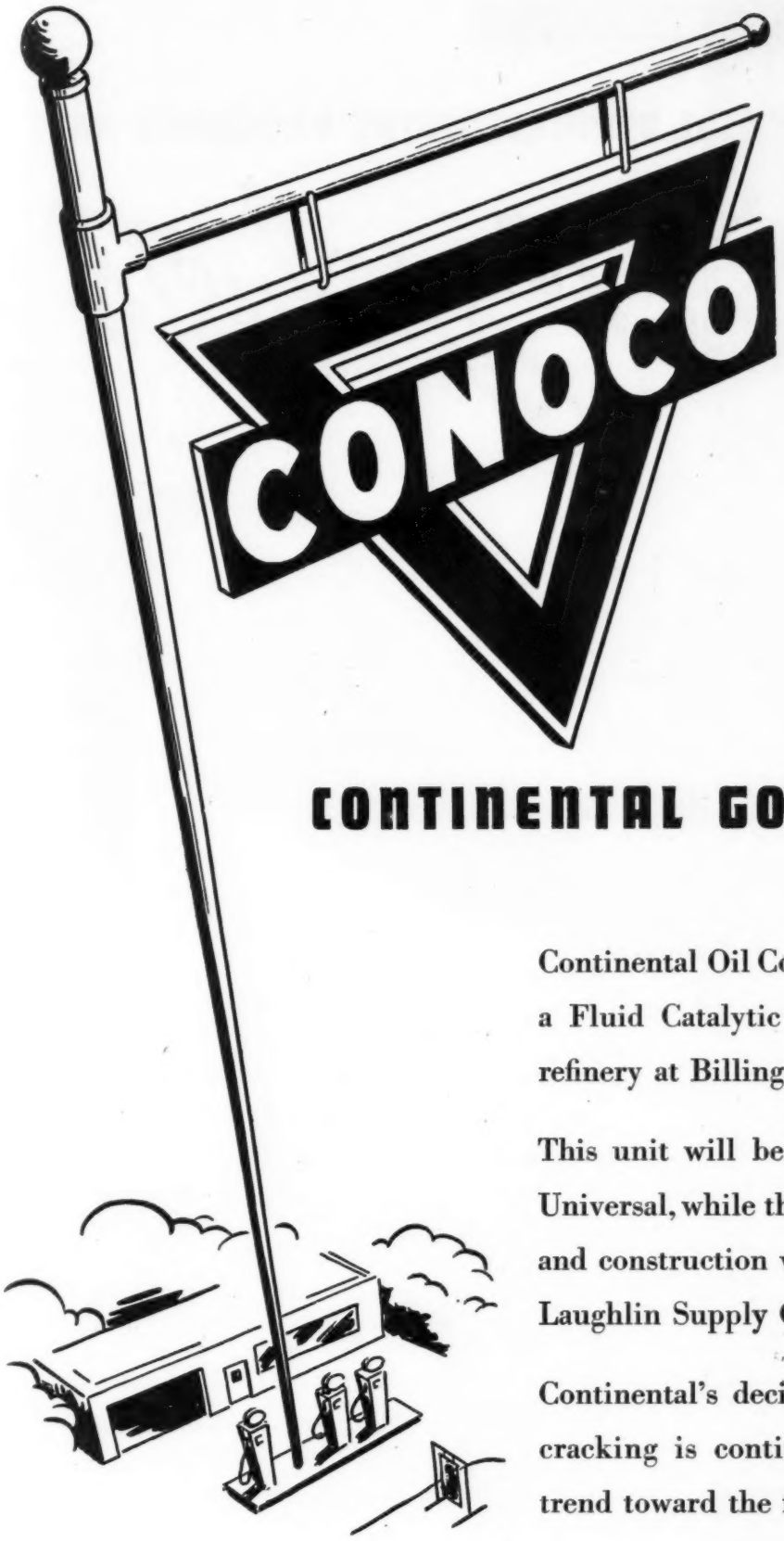
The possibility remains that the heart cut of constant composition might be an iso-octane azeotrope containing fractional amounts of some olefin or alcohol. Using −107.311° C. as the freezing point of pure iso-octane the original sample would calculate as 99.49% pure, and the heart cut as 99.74% corresponding to 0.26% impurity. The techniques of infrared and chemical inspection used on the present sample are such that as much as 0.1% of either type of compound should have been detectable, but no evidence of impurity was found in the heart cuts.

Selective adsorption on silica gel is a powerful tool for removing polar impurities from a paraffin hydrocarbon, and it should remove completely any impurity capable of forming an azeotrope. It will remove other impurities to some extent, but this tendency is of a different order of magnitude. Silica gel percolation of the heart cut fraction changed the freezing point from −107.377 to −107.372, indicating some improvement but no major change. Percolation of the bottoms removed all evidences of toluene and diisobutylene, giving a product which was essentially pure iso-octane by infrared analysis. It is concluded that the material in the heart cut is not an azeotrope and that the analysis given above accounts for the impurities present in any significant amount.

These analyses indicate that the total impurities present in certified iso-octane, Batch No. 2, amount to 0.38 mol %. Small amounts of material of still higher purity may be prepared from this source as needed. Percolation of this iso-octane through silica gel changes its freezing point from −107.438 to −107.406° C. This is equivalent to increasing the purity to 99.75 mol %. An efficient topping operation at high reflux ratio to remove the first 6%, followed by percolating the bottoms through silica gel, gives a product having a freezing point of −107.368° C. which is equivalent to 99.90% purity.

TABLE 9—Scheme of Analysis for Certified Iso-octane, Batch No. 2, 90-Plate Distillation, 890 ml. Charge

Reflux Ratio	Cut No.	Vol. %	Ref. In. Increment	Infrared % 2,2,4	Infrared Impurities	Fr. Pt. °C. (Melt. Curve)	Purity Mol. %	Other Tests
700:1	1	0.25	−0.00021	88	10% 2,3-dmp 2% (?)			
	2	"	−0.00015	92	8% 2,3			
	3	"		96	4% 2,3			
	4	"	−0.00003	98	2% 2,3			
	5	"						
300:1	6	"						
	7	"						
	8	"	−0.00001			−108.144 from initial 3.92% on duplicate rerun	96.82	Water Content after F.P.=0.01 Wt. %
	9	"						
	10	"						
	11	"						
	12	"	−0.00000					
	13	"						
	14	"						
	15	"						
100:1	16	5.8	−0.00000	pure		−107.397	99.79	
	17	"						
	18	"						
	19	"						
	20	"	0.00000			−107.377	99.87	
	21	"						
	22	"						
	23	"						
	24	"	Standard					
	25	"						
	26	"						
	27	"						
	28	"	0.00000					
	29	"						
	30	"		pure		−107.377	99.87	
	31	0.6						
	32	"	+0.00000					
	33	"						
	34	"	+0.00000					
	35	"	+0.00001	100	trace			
Bottoms		3.7	+0.00066	ca 95	0.06% Tol. 2,4,4-tmp=1 2,4,4-tmp=2	50/50 blend with cut 20 −107.799	98.19	Tol. by Raman=0.07% Br. No.=1.5
Original		100.0	−0.00000	pure	no evidence	−107.438	99.62	Water, 0.006 wt. %



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Contact Finishing of Lube Oil Stocks *

Improvements in the contact filtration method for finishing lubricating oil stocks, largely the continuous operating feature, are bringing about greater use of this method. Contacting temperatures applicable vary from room temperature to 600° F. Stripping under vacuum promotes the decolorizing action of the adsorbent and provides other advantages. Use of a continuous rotary precoat type filter reduces the oil content of the spent adsorbent as compared with that when a leaf type batch filter is used. Automatic devices proportion the adsorbent continuously to the oil stream.

THE contract filtration process is used by petroleum refiners to economically and efficiently remove resins, color bodies, acids, and other undesirable constituents from a wide variety of acid treated or solvent refined stocks to produce high quality lubricating oil, waxes, and specialty products.

The advantages of the contact process over other methods of finishing are even greater now than in past years, due to improvements in equipment, automatic controls and in the activated adsorbent employed, which account for its acceptance as the finishing method to be employed in many new lube oil expansion programs now under way.

The contact filtration process is simple and direct. It consists of heating a homogeneous mixture of adsorbent and lube stocks under optimum conditions and separating the spent adsorbent from the oil. In the process flow the adsorbent is added to the oil charge by means of automatic proportioning equipment. The slurry enters a pipe still heater where the ultimate contact temperature is obtained in a once-through operation. From the heater, the slurry enters a baffled vacuum stripping tower where any desired level can be maintained thereby regulating the time of contact.

Facilitating Stripping Action

Steam or inert gas is usually passed up the tower to facilitate the stripping action. The slurry is drawn continuously from the bottom of the tower, passed through heat exchangers (to heat the fresh oil charge) and pumped to a continuous precoat rotary filter. The filtered oil may be charged to a high vacuum stripper to furnish additional control of flash, odor, oxidation, and emulsion characteristics. The final operation is to filter in a blotter press as a "polishing" step before the oil is transferred to storage.

With the contact filtration process, operating conditions control the quality of the finished oil and each stock has specific treating conditions and adsorbent dosages which are optimum. The time of contact usually varies inversely with the treating temperature. The optimum contacting temperature varies from room temperature to 600° F. Normally neutrals should be treated at low temperatures (165° F. to 300° F.) while bright stocks require temperatures of 500° F. to 550° F. For lubricating oil a general rule to follow for contact temperatures would be 30° F. to 35° F. below the flash point of the oil, although exceptions exist. For crystalline wax, white oil and insulating oil the lowest possible treating temperature usually gives the best results. A temperature of 300° F. to 325° F. minimum is recommended for finishing microcrystalline wax from solvent treated bright stock origin.

Selection of Stripping Media

The selection of the proper stripping media to be used should be determined by plant operation after indicative results have been obtained in the laboratory. Stripping during contacting may be accomplished with flue gas, steam, vacuum, or a combination of steam and vacuum; superheated steam is recommended. Steam finds the wider application in commercial operations. On certain stocks air has given results superior to either steam or inert gas. Vacuum during the stripping operation promotes the decolorizing action of the adsorbent by acting as an auxiliary agent and reducing the quantity of steam or inert gas required. The use of high vacuum stripping as a final step after the contact operation is highly recommended, as it invariably improves the odor of the oil and on certan stocks improves oxidation and emulsion characteristics.

Sufficient agitation should be employed to keep the adsorbent in continuous suspension. This is especially true in low temperature contacting operations where laboratory and plant results have shown that poor agitation may increase the amount of adsorbent necessary to finish an oil.

The success of the contact filtration process is dependent upon the proper design and function of certain equipment. The filter is probably the most important item and the widespread acceptance of the process dates from the marketing of the first continuous rotary precoat type filter. This unit requires a minimum of manpower for operation and disposal of spent adsorbent. The oil content of the spent adsorbent is reduced nearly one-half of that obtained when using the leaf type batch filter.

The heater for the oil-absorbent slurry should be designed so that high skin temperatures are not developed in the furnace, thus avoiding cracking of the oil during heating. The service records of pipe stills in this type operation are good, and there are units which have been in continuous use for several years without a major overhaul.

Pumps for Handling Slurry

The slurry pumps are the cause of considerable difficulty in the contact operation. The reciprocal steam piston pump is recommended; however, many units successfully utilize centrifugal pumps for this service. In any case, the pump should be oversize for the amount of oil to be pumped so that it can be operated at reduced speed, thereby decreasing erosion by the adsorbent. Spare hot-roll slurry pumps should always be installed to allow down-time for pump repair.

Automatic handling devices have nearly eliminated the requirement of manpower for the introduction of the adsorbent into the charge oil. Either mechanical or pneumatic conveying systems may be used for transferring adsorbents from bulk cars to the primary storage bins, thence to the feed bin. A gravimetric weight feeding machine has been developed and is widely used commercially, which adds the adsorbent continuously into the oil stream in any desired quantity.

The development of this machine has been a great boon to the processing since, in addition to savings in manpower, it allows more uniform mixing of the total oil and activated adsorbent charge. Some operators mix the total adsorbent dosage with the total oil charge in a single operation. Others mix the total adsorbent with a percentage of the oil charge and blend the slurry with the total oil charge just before the oil enters the furnace. Either of these methods gives equally good results.

The contact tower should be designed to provide adequate stripping and agi-

* Material for this article was supplied by the Filtrol Corp., Los Angeles.

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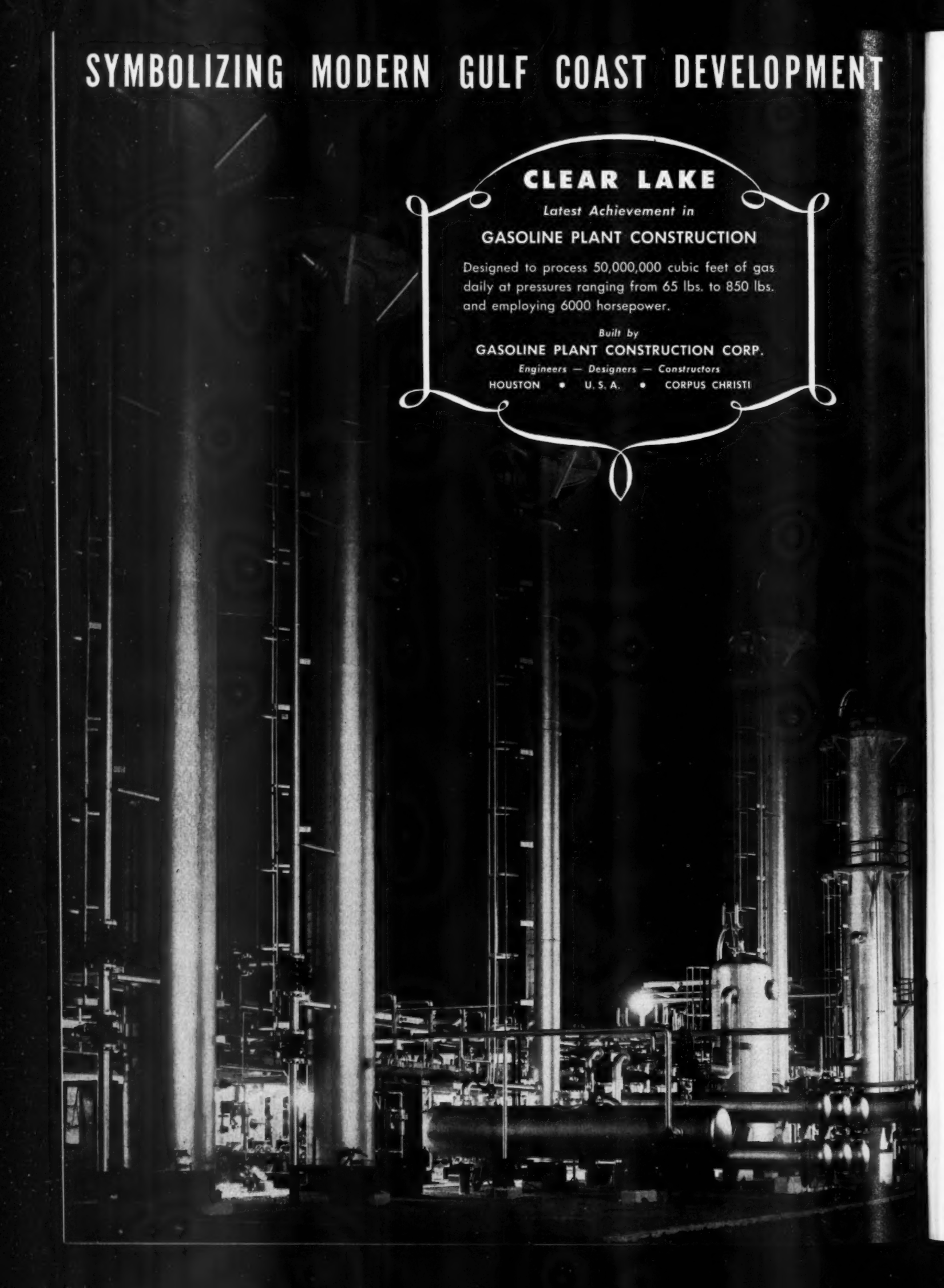

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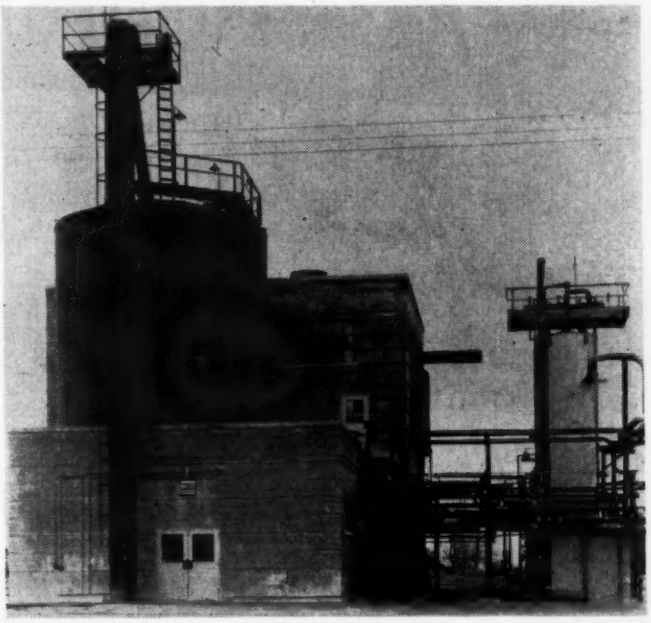
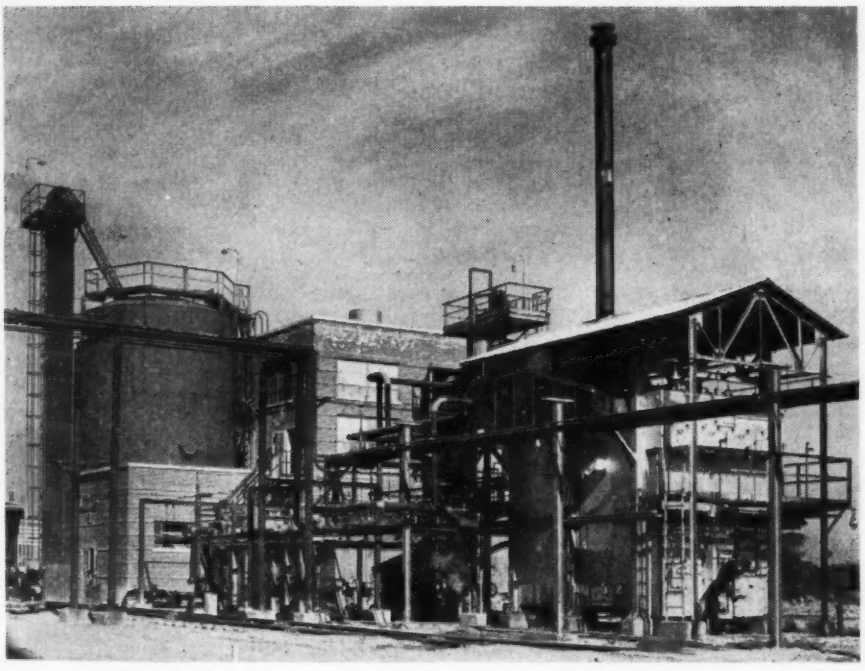


Two views of a typical contact filtration plant for finishing lubricating oils, waxes and specialty products. Photo at right shows the clay unloading elevator and storage bin. Photo below shows, in addition, the furnace where the oil and clay slurry are heated prior to being charged to the contact tower

Photo at right courtesy the Filtrol Corp.
Photo below courtesy British-American Oil Co.



tation sufficient to keep the adsorbent in uniform suspension. The high vacuum stripping tower after filtration should be equipped with splash plates or other internal dispersing equipment designed to allow maximum stripping effects so that the oil may be freed of moisture, objectionable odor and cracked products. This tower may be designed so that most of the necessary stripping can be obtained in this unit, but its primary purpose is to remove by fractionation small amounts of undesirable hydrocarbons that boil slightly below the lightest fraction of the oil being contacted.

The use of the contact filtration process is continually expanding to new applications and following is a résumé of stocks which are being finished on a commercial scale in this manner.

Solvent refined, acid treated, and straight distillate and residual stocks from paraffin, mixed, and naphthene base crudes are being finished in large volume to high quality motor and industrial oils, in conjunction with the Duo-Sol, Furfural, Phenol, Edeleanu, and Propane Deasphalting processes. Contact filtration is also recommended for finishing turbine and transformer oils to the rigid specifications demanded by the U. S. and the British Governmental agencies. Medicinal and technical grade white oils and crystalline waxes are finished in limited quantities.

In the U. S., because of previously developed technique and existing plant facilities the larger volume of specialty products is finished by the percolation method. However, in Europe these stocks are finished almost exclusively by the contact filtration method. Microcrystalline wax from solvent treated bright stocks of tank bottom origin is finished very successfully with adsorbent contacting. In fact, due to the heavy dosage of fullers earth required for bleaching these stocks the use of an activated earth is almost imperative. Lubricating oil additives and petroleum resins are bleached successfully with adsorbent contacting. The decolorizing of such materials is being employed on an expanding scale in both commercial plants and in laboratory experimental work.

The steady growth of the adsorbent contacting process may be attributed to the following advantages obtained by this method of finishing:

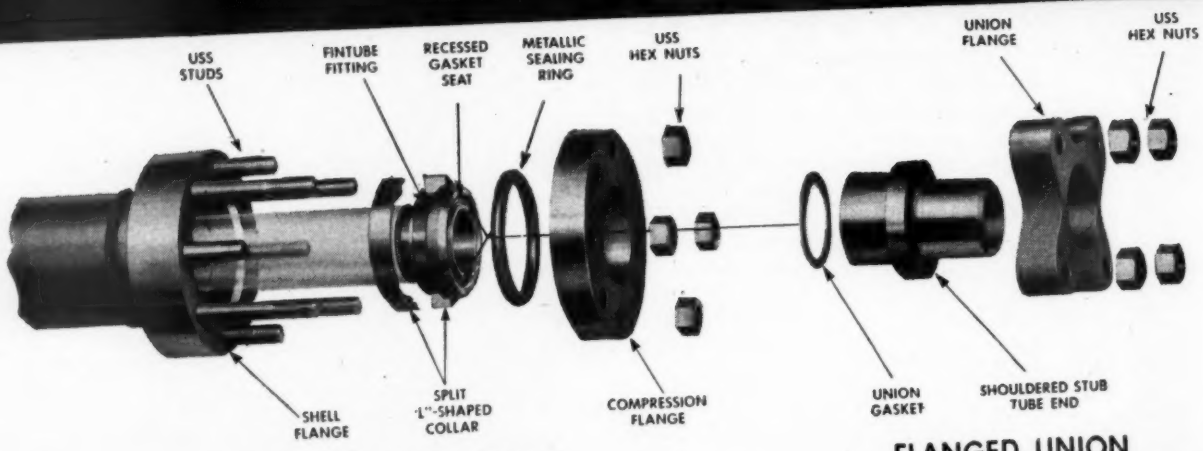
(a) Utilizes a single constant quality chemical which is readily available to all refiners at a low per barrel cost.

(b) Employs a single treating plant and hence, a minimum capital investment to provide for long continuous runs on a single stock, or a blocked out operation on a number of stocks.

(c) Operates efficiently on many charge stocks with a wide variation of optimum contacting conditions. In one refinery, a single contact plant is being used to finish two full lines of SAE grade motor oils from low and high V. I. stocks as well as bright stocks, crystalline wax, and several industrial oils.

(d) Lower labor cost—the modern contact plant can be operated efficiently with two men per shift whereas in other adsorbent processing plants of the same approximate finished oil capacity up to 8 men per shift are required.

THIS EFFICIENT HEAD END ASSEMBLY



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MANUFACTURERS OF BROWN RESISTANCE WELDED INTEGRALLY BONDED ONE-PIECE FINTUBES, AND FINTUBE HEAT TRANSFER PRODUCTS

Extraction Plants Replace Gas Flares

Removal of Liquid Hydrocarbons from Casinghead Gas for Use In Motor Fuels, as LPG and in Chemical Manufacture Is One Phase in Conservation of Gas from Oil Fields, as Voluntarily Practiced by Humble and Other Oil Companies in Southwest

MILLIONS of dollars are being voluntarily expended every year by oil companies of the southwest in extracting liquid hydrocarbons from the natural gas produced along with the oil from oil wells, and in making further economic utilization of the gas, in a most practical form of conservation of the nation's petroleum resources.

Of course, there is still considerable "flare" gas which it is not economic to use at present. Where the quantity is small, where it is considerably contaminated with carbon dioxide, hydrogen sulfide or other impurities, or where the limited production is remote from gas lines to take it to consuming markets, the casinghead gas produced with the oil often cannot be saved. It may not even be feasible to return such gas to the producing formation. The amount of casinghead gas that cannot be conserved, however, is constantly decreasing.

One of the notable examples of this voluntary type of conservation is Humble Oil & Refining Co., Houston, a pioneer in this work. Before the war, while a large producer of crude oil, it owned only eight natural gasoline plants and was part owner of another. It had many gas repressuring projects in operation, following the first experimental installation of this type of plant in 1930. The war temporarily stopped further expansion of Humble's gas conservation, to allow the company to put all its efforts on facilities to make aviation gasoline and other essential wartime products.

When the war ended, the company, in resuming its gas conservation program, first surveyed all its flared casinghead gas production to determine what portion might be economically utilized. The survey revealed that Humble was producing approximately 408 million cubic feet of casinghead gas daily, 210 MMcf (52%) of which was being flared and 198 MMcf conserved. It has since initiated or completed projects in 22 fields which will increase total gas utilization by another 110 MMcf. Twenty-seven additional projects still in the planning stages are expected to increase utilization another 69 MMcf.

When all the projects now under consideration, construction, or in early operation stages, are finished, Humble's casinghead gas utilization will have been increased to 377 MMcf, or about 92% of the quantity being flared in the fall of 1945. Of the remaining 31 MMcf, 7.5 MMcf is in fields in which Humble is not the major producer but is willing to cooperate in a feasible utilization program. Fields in initial development stages, where accurate data on the amount of gas to be available is yet indeterminate, account for another 7 MMcf. The remaining 16.5 MMcf is from fields in which the very small quantity, high sulfur content, remote location or other factors make useful utilization impractical from an economic standpoint.

When projects now definitely planned are completed, Humble will have spent about $10,000,000 on gas conservation facilities since the end of World War II. Plans now under consideration will increase this amount substantially.

These projects include natural gasoline plants, cycling, pressure maintenance, repressuring, gas lifting of oil, fuel for employee housing camps, and other uses. In all of them the company has followed its policy of producing oil with a minimum amount of gas and, where gas is produced, of marketing, consuming or returning it to the formation, after the liquid hydrocarbons have been extracted, if this latter operation is practicable.

Two natural gasoline plants, Clear Lake and Anahuac, have been completed this year at a combined cost of $4,750,000. Another, in the Tomoconnor field to cost $3,000,000, and 50% Humble-owned, is now under construction with completion scheduled for late 1947. Clear Lake will extract 41,000 gal. per day of natural gasoline, and Anahuac 36,000; Tomoconnor is expected to produce 60,000 gal. per day. Combined, these plants will process approximately 120 MMcf

Fig. 1—General view of Humble's Clear Lake natural gasoline plant. Major equipment visible, left to right, deethanizer, gasoline still, low and high pressure reabsorbers and the two primary absorbers. In front, on the ground, are small gas scrubbers. In foreground are the meter runs on low-pressure inlet gas to compressors and high pressure residue gas lines. Large vertical vessel at left is a steam condensate tank; horizontal vessel in rear is raw gasoline surge tank

Fig. 2—Portion of the compressor house at Clear Lake. Compressor horsepower totaling 4000 h.p. and a 200 h.p. recompressor handles 25 MMcf of low-pressure gas and plant recompression requirements

daily of previously flared casinghead gas.

Clear Lake Plant

The Clear Lake plant compressor section went on stream last March, before construction of the distillation system was complete. It is an example of good economics in gas utilization. Fig. 1 is a general view of the process area.

This project was initiated late in 1945 to provide a means of compressing and processing all the low-pressure casinghead gas from nearby oil fields then being flared and also to process gas well gas which previously had been passing directly into a gas transmission system (Tomball) supplying the Baytown refinery with plant fuel. A small low-pressure gasoline plant at Dickinson was dismantled and replaced with a compressor installation to boost its former intake into the high pressure Clear Lake plant.

Clear Lake is designed to handle a total of 50 MMcf of gas per day, of which 25 MMcf is received at 810 psi. from gas wells and Dickinson, and 25 MMcf enters the plant intake at approximately 60 psi. Breakdown of gas receipts from the field is shown in Table 1. The two streams are kept separate as noted during processing.

Field Gas System

Gas is delivered from the Webster field to Clear Lake through an 18-in. line Field separators operate at an approximate pressure of 75 psi. An emergency pressure relief regulator venting to a flare is installed at the plant to prevent excessive field separator pressures and flaring in the field in event the compressor unit is shut down. Field separators in the East Clear Lake field are operated at sufficient pressure to introduce gas into the plant at a pressure of 60 psi.

Construction work at the Dickinson plant involved installation of three 600 hp compressors and necessary equipment for compressing the oil well gas from the Dickinson-Gillock field. (Additional facilities now are contemplated to increase the volume handled.) Compressors take suction at 35 psi. from field separators and discharge through three compression stages at a final pressure of 1000 psi. In distinction to Webster and East Clear Lake gas however, Dickinson gas enters the plant with the gas well gas.

The gas well at Hall's Bayou and the wells in the Dickinson field are produced sufficiently to maintain the load of the Clear Lake plant and to meet necessary sales gas demands. This gas is delivered to the plant at 810 psi.

Plant Description

The Clear Lake plant is largely a conventional, two-stage oil absorption type, equipped with fractionation to supply 41,000 gal. per day of de-ethanized natural gasoline containing 55% of the propane, 99% of the butanes and 100% of the pentanes and heavier originally in the gas. The gas is lean, especially in C_5 and heavier hydrocarbons. This is the result of operating field separators at 75 psi., which causes portions of the

TABLE 1—Gas Receipts at Clear Lake Plant

Field	Volume Mcf/day
Oil Well Gas	
Webster	20,000
East Clear Lake	5,000
Dickinson-Gillock*	6,000
Total	31,000
Gas Well Gas	
Hall's Bayou	4,000
Dickinson	15,000
Total	19,000
GRAND TOTAL	50,000

* Enters plant processing system with gas well gas.

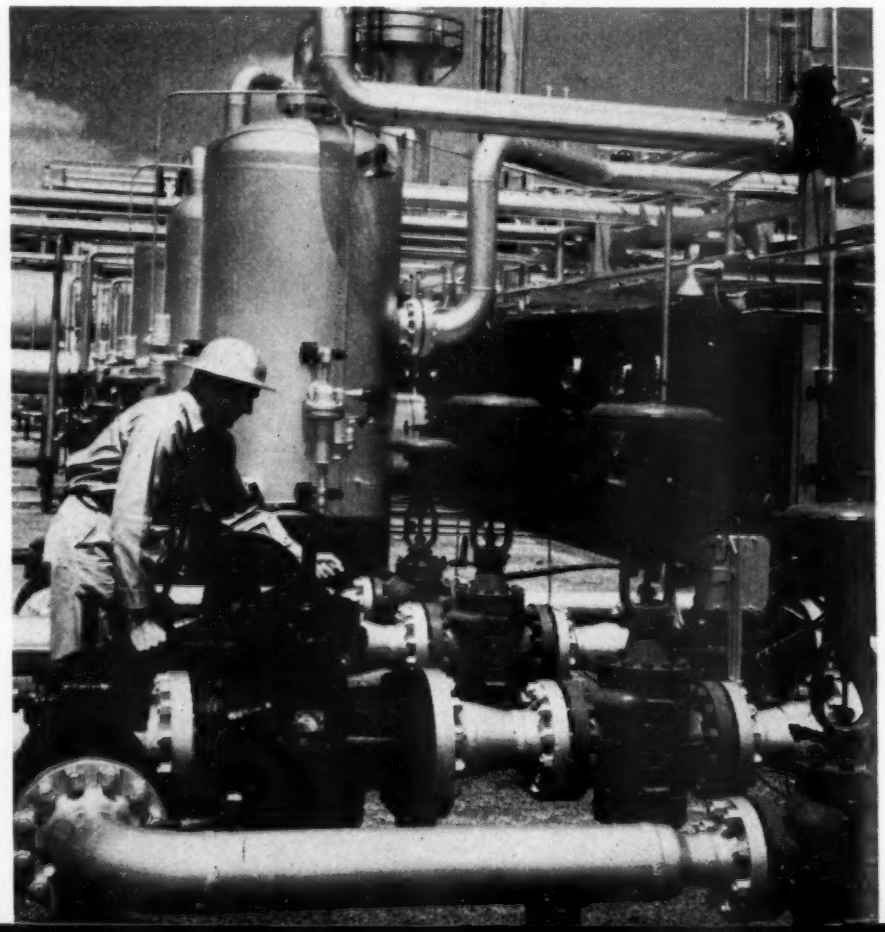

Fig. 3—Placing a regulator setting on dry residue gas line at Clear Lake. Short vertical vessels behind operator are inlet scrubbers

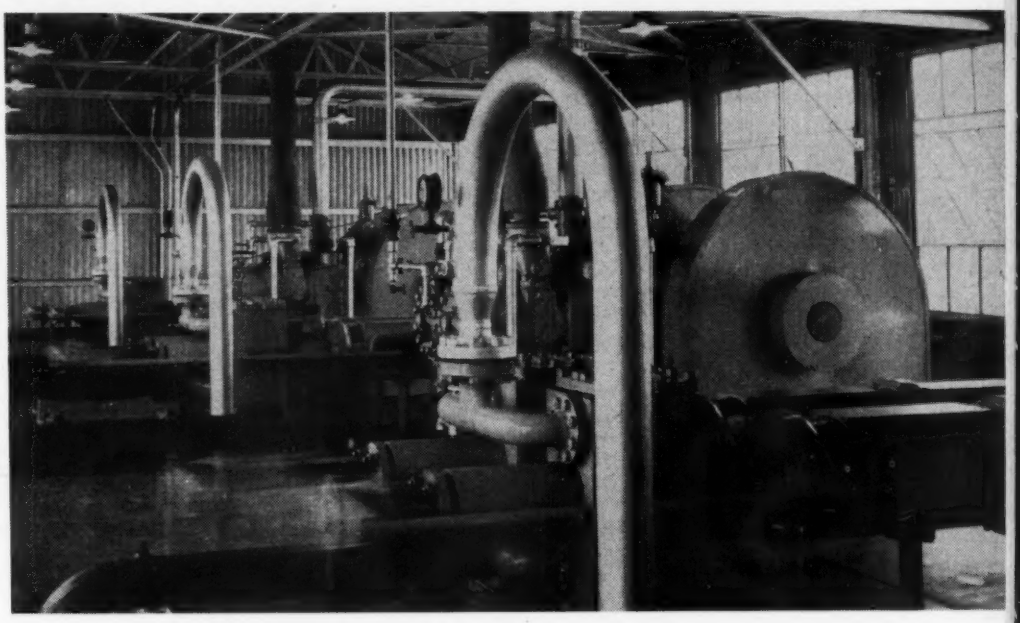

Fig. 4—Two high pressure pumps each supply individual high pressure absorbers with 240 g.p.m. of lean oil

heavier fractions to remain in the crude. In fact, Clear Lake would not pay out financially on the basis of liquid product recovery alone under present operations. Payout actually is based in addition on expected revenue from residue gas sales. Elimination of gas flaring—conservation—is also a factor.

No facilities are provided to produce propane or LPG mixtures; the entire liquid product is pumped 12 miles by pipeline to Baytown. Care has been taken to design the Clear Lake plant for a minimum of fuel consumption in order to provide the maximum amount of residue gas for the transmission system. Extensive use is made of reabsorption facilities to hold C_3-C_4 losses to designed maxima. Also, by suitable adjustment of de-ethanizer operation, a gaseous product containing 2.5% methane, 25% ethane and 72.5% propane can be supplied for chemical synthesis.

Low-pressure oil well gas enters the plant through a meter run, then an inlet gas separator and finally the compressor suction header. Two stages of compression boost the gas from its initial 60 psi. to 810 psi. (Fig. 2). Cooled, compressed gas then enters one of the two high pressure absorbers. Residue gas from the absorber passes directly to the gas transmission line through a sales meter run. High pressure gas well gas enters the plant at 810 psi. through a meter run and separator, thence directly to the second high-pressure absorber and finally the transmission line. Fig. 3 shows a portion of this section of the plant.

Lean absorption oil is pumped over each of the main absorbers by separate reciprocating pumps, Fig. 4, at a rate of 220 to 240 g.p.m. The rich oil streams combine and pass into a flash tank from which the flashed rich oil continues on through heat exchangers, preheaters and the primary stripping still. Vapors at the flash pressure of 250 psi. enter a reabsorber, the residue gas from which is utilized as plant fuel.

Flashed rich oil is only partially stripped in the rich oil stripper, overhead from which it is again reabsorbed (at 175 psi.) in order to retain the desired quantity of propanes and butanes, while eliminating the major portion of the ethane and methane.

Stripping of the rich oil is completed in the gasoline still, which operates at 60 psi. and 415° F. Also going to the gasoline still is the rich oil from the second reabsorber, after passing through separate heat exchangers and a preheater. The main still base serves as a surge space and from here the hot lean oil passes through two sets of heat exchangers, the oil coolers and into the oil pump suctions for recirculation.

Still overhead vapors are passed through a primary and secondary set of condensers and receivers. Liquid from the first receiver supplies reflux to the column. Vapors and liquid from the secondary receiver are taken off separately, vapors being recompressed to 300 psi. and the whole recombined in a raw make tank after water cooling to 90° F. This material is de-ethanizer charge.

The de-ethanizer operates at 475 psi. and 280° F. reboiler temperature. Overhead product, predominantly ethane with a small quantity of methane and propane under present operation, also is used for plant fuel. Kettle product is an ethane-free gasoline of about 95-lb. Reid vapor pressure and consists of 32% propane, 38% butanes and 30% pentanes and heavier.

Plant Utilities

The compressor section of the plant consists of four 1000 hp two-cycle gas engine driven compressors, each supplied with two low-stage and two high-stage cylinders. One end of the compressor building, shown in Fig. 2, also houses the 200 hp recompressor. Cooling water for the power and compressor cylinders is supplied from a closed system containing corrosion-inhibited condensed steam. Temperature for the power cylinder coolant is automatically controlled for each unit. All large gas engines in the plant are provided with automatic shut-down devices in the event of low lubricant pressure or water failure. A monorail and bridge-type crane is supported from the building members so that any part of the compressors can be made readily accessible for maintenance.

The steam generating facilities consist of three 150 hp 350-lb. working pressure oil-country boilers which supply 25,000 lbs. per hour of saturated steam at 300 psi. Three separate steam systems are utilized: The 300 psi system serves all plant preheaters, reheaters and reboilers; a 250 psi system which furnishes power for reciprocating pumps, and a 60 psi system (exhaust from the 250 psi system) which supplies stripping steam for the main still. All low pressure condensed steam which has become contaminated with oil is accumulated in an oil-skimming tank, from which it is pumped back to the deaerating feedwater heater. High pressure condensate is partially cooled and then reinjected into the deaerating heater. For these reasons, the boilers require only 8-10% raw makeup and feedwater is automatically treated with chemical through a proportioning pump before entering the boiler.

The entire plant and camp electrical demand is supplied by a generation plant which consists of three 250 K.w., 440 v. 60 cycle gas engine driven generator sets. Fig. 5 shows a portion of this equipment. The switchboard is provided with automatic synchronizing devices for simplicity of operation in addition to the conventional breakers, indicators and meters. In addition to the low oil pressure and high water temperature shutdowns for the generator drivers, an alarm system has been installed to sound a horn in the event of a drop in oil pressure or if water temperature starts to increase, thus warning the operator a shutdown is imminent.

An ample supply of deep well

water, 3.5 gr. per gallon hardness, is available from two wells drilled in the plant yard. One is provided with an electrically-driven pump while the other has a combination motor-and-gasoline engine drive. The cooling tower is an induced-draft type with rated capacity of 7000 g.p.m., cooling from 100° to 85°F. One hundred horsepower deep well type motor driven pumps supply circulation to the plant. Dissolved solids content is maintained at not more than four times the original concentration with a continuously-metered purge. Bacteria control is obtained by continuous chlorination.

Provision for Safety

Safety considerations have not been left out. Six automatic shutdown stations are scattered about the plant. Each contains a single valve which, when opened, transmits gas pressure to various piston-operated block valves and motor valves. These in turn block inlet high- and low-pressure gas streams, outlet high-pressure gas from both main absorbers, open a vent valve dumping high-pressure residue gas to the flare pit, block fuel gas to compressors, generator engines and lean oil pump engines, shut off gas to boilers and open snuffing steam lines in boiler fireboxes. The plant can be almost completely shut down in seconds.

All compressor suction scrubbers are provided with low level alarms and automatic liquid dump valves; if liquid should build up to a still higher point, a float-operated switch is actuated which grounds out the compressor magnetos. All absorber residue gas scrubbers are provided with an automatic liquid dump and alarm in the event of oil accumulation.

Anahuac Plant

Humble's latest natural gasoline plant to come on-stream is the Anahuac plant, serving the field of that name. Initial operations commenced Aug. 10. It is rated at 36,000 gal. per day of liquid hydrocarbons from 45 MMcf of a casinghead gas somewhat richer than that processed at Clear Lake. The plant product consists of a de-ethanized natural gasoline that will be moved to the Baytown refinery by pipeline.

Inlet gas from Anahuac separators is received at 45 to 50 psi, although this may be raised later to 75 or 80 psi. This gas then is compressed in two stages to 810 psi at which pressure it enters the high pressure absorbers. The residue gas from these absorbers then is passed into Humble's Tomball natural gas system for transmission to consumers. Design calls for the recovery of 22% propane, 85% butane and 99% pentanes and heavier.

Justification of the plant, according to Humble management, also is on the basis of residue gas sale and products recovery, neither of which will be sufficient alone to pay out the plant. Cost is $2,500,000.

Other Projects Completed

Thirteen other projects, which did not involve gasoline plant construction, also have been completed in Humble's gas conservation program, six in Louisiana and the balance in Texas.

In the Amelia field facilities were installed for injecting into the formation 2.3 MMcf of previously vented casinghead gas at a cost of $165,000. Reservoir studies had indicated that the very strong water drive was forcing the thin layer of oil into the gas cap, which would result in decreased oil recovery.

Facilities costing $500,000 to handle 8 MMcf of flared gas in the North Katy field were added at the Katy cycling plant operated by Humble. This gas now is processed for a total additional recovery of 6000 gal. per day including propane and butanes. Residue gas is sold to Tennessee Gas & Transmission Co.

In the Lovell Lake field facilities were provided to inject 2 MMcf of previously flared casinghead gas into the lower Frio sand; operations began in October, 1946. In addition, 4.7 MMcf now is gathered and compressed for disposal into the Tomball gas system. Total cost of the project was $170,000.

The North Tepetate, La., installation, consisting of individual high-pressure separators and a gathering system, handles about 500 MMcf of casinghead and 18 MMcf of gas well gas. A dehydration unit also has been installed to remove excess water before delivery to Interstate Natural Gas Co.

About 4.3 MMcf of casinghead gas now produced with oil in the Chalkley (La.) field, is being returned to the formation, there being no sale for this gas.

The Roanoke, La. gathering system and compressors cost $78,000 to compress 1.7 MMcf of previously flared casinghead into the transmission line of United Gas Pipe Line Co.

The North Crowley, La. project is handling about 7.25 MMcf through a compressor station and 9-mile lateral which connects to Humble's Pecan Island line. The work cost approximately $390,000 to supply the gas eventually to Interstate.

At Good Hope, La., high-pressure separators and a gas gathering system were installed to furnish about 1 MMcf per day to United Gas. A similar installation was made in the Paradis field to supply United with another 2 MMcf.

With similar operations under study or construction by other operators it will not be many years until gas flares indeed will be "news" in Texas and Louisiana.

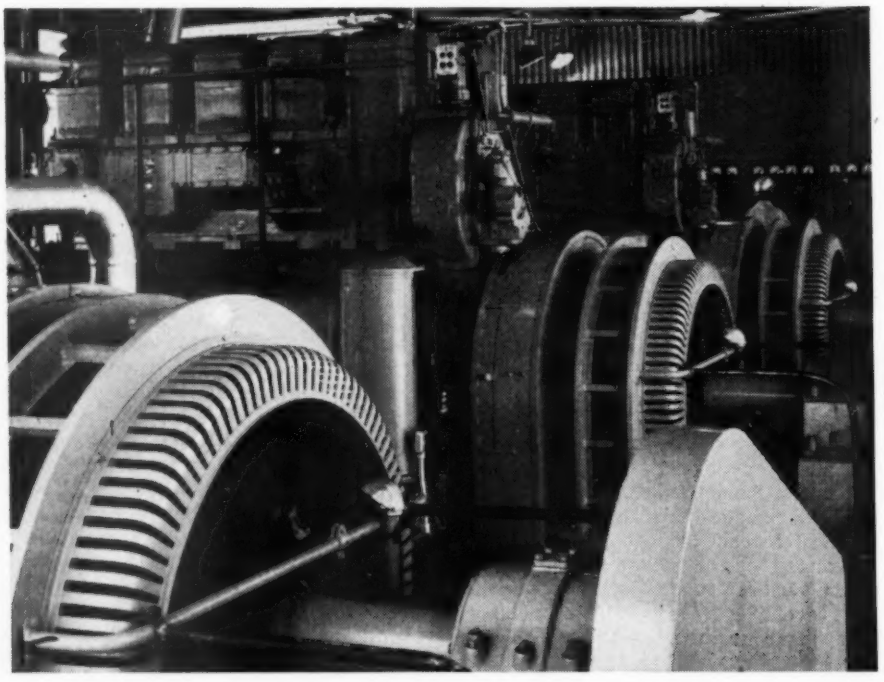

Fig. 5—Power generating equipment at Humble's Clear Lake plant. These three 250 K.w. gas engine-driven generators supply plant demand and employee-camp requirements

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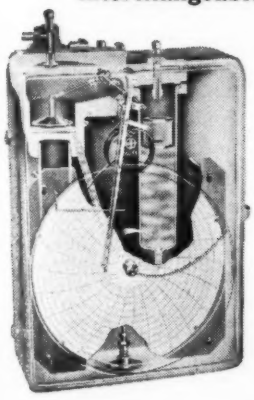

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STRIPPING AND RECTIFYING COLUMNS FOR TERNARY DISTILLATION

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Graphical methods are demonstrated as effective means for determining equilibrium plates in distillation columns involving ternary mixtures.

Two examples are given by the authors in developing a description of the technique and explaining the calculations. One is for a stripping column with a feed mixture of 40 mole-% pentane, 30 mole-% hexane, and balance heptane, to be stripped to a heptane content of 60 mole-%. The other is a rectifying column whose feed mixture contains the same components as in the first example to be distilled to a product containing 70 mole-% pentane.

THE operation of stripping columns to which the stripping vapor is introduced from a still has been considered previously(1) and illustrated by calculations with binary mixtures. The following example shows the calculation of the number of equilibrium plates in a stripping column when fractionating a ternary mixture.

Consider the following problem. A feed mixture containing 40 mole-% pentane, 30 mole-% hexane, balance heptane is to be stripped to a heptane content of 60 mole-%. The feed is to be introduced as a saturated liquid (at its bubble point) to the top plate of a stripping column operated at 15 psia. It is desired to determine the relation between the "boil-up" ratio and the number of equilibrium stages required. By "boil-up" ratio is meant the ratio of the rate at which vapor leaves the still to the rate at which the bottom product is withdrawn.

A stripping column is illustrated in Fig. 1a, and the composition diagram in Fig. 1b for the case where the "boil-up" ratio, V_s/B, equals infinity corresponding to a total reflux operation requiring a minimum number of stages.

When V_s/B is equal to infinity, the amount of bottom product B will be equal to zero and by a material balance the composition and quantity of the overhead product V_1 will be equal to the composition and quantity of the feed. Thus, the point F in Fig. 1b represents both the feed and the overhead product.

The liquid leaving the top equilibrium plate is in equilibrium with the overhead vapor V_1 and is represented by a point L_1 at the end of the tie line from V_1. The quantity and composition of the vapor V_2 leaving the second equilibrium plate is equal to the quantity and composition of the liquid L_1 leaving the top plate since the difference between these streams by a material balance is equal to stream B or zero. Thus point L_1 also represents the stream V_2. The liquid leaving the second plate L_2, therefore, lies on the equilibrium tie line from point L_1.

Plate to plate calculations are continued in this manner until a liquid composition is attained which contains more than the specified amount of heptane. The fractional equilibrium stage is estimated as in binary calculations and in Fig. 1b the number of equilibrium stages required is seen to be 0.95. The separation required in this case is easy, and only the one stage needs to be calculated.

When the ratio V_s/B is some finite value such as 2.0 the quantity of the bottom product, B, is fixed since under constant molal overflow conditions the quantity of over-

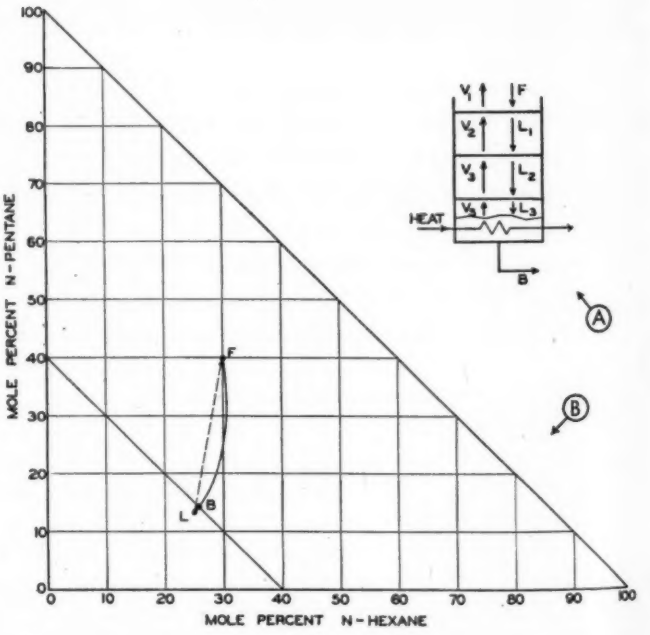

Fig. 1—(A)—Diagrammatic stripping column. (B)—Composition diagram of a stripping column at infinite "boil-up" ratio

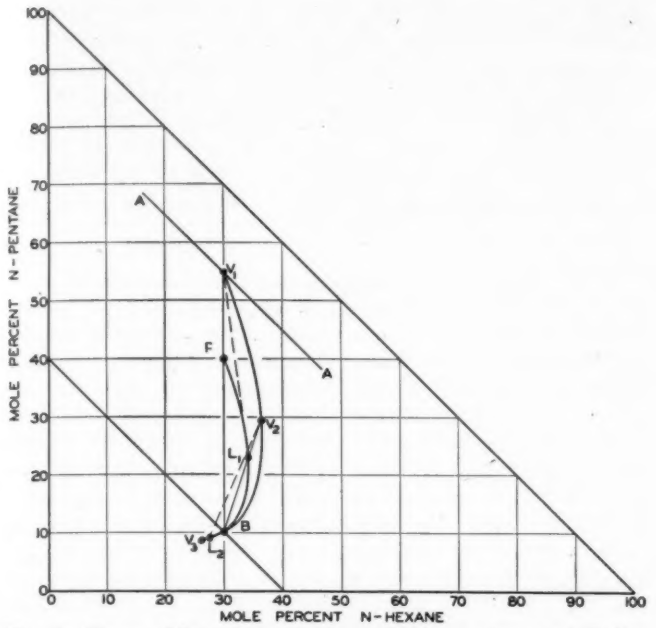

Fig. 2—Composition diagram for stripping column at finite "boil-up" ratio

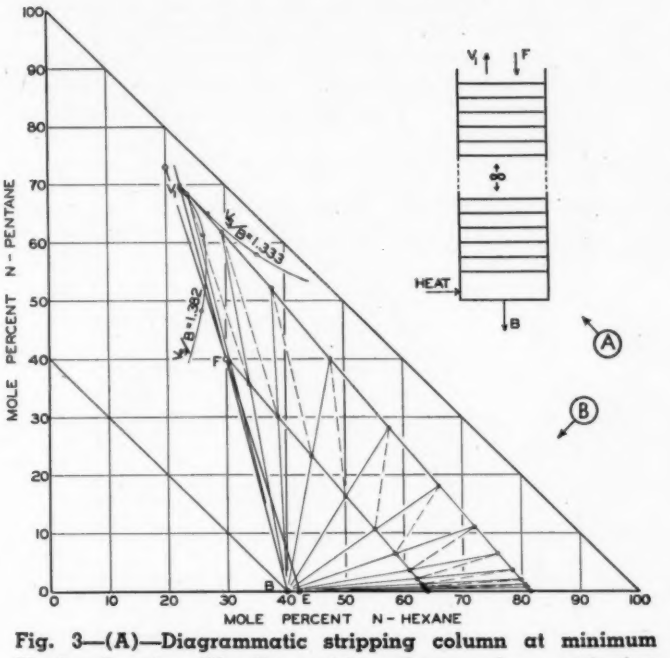

Fig. 3—(A)—Diagrammatic stripping column at minimum "boil-up" ratio. (B)—Composition diagram for a stripping column at minimum "boil-up" ratio

head product V_1 is equal to the quantity of vapor leaving the still, that is,

$$F = V_1 + B = 1.0$$
$$V_s/B = V_1/B = 2.0 \text{ or } V_1 = 2.0B$$
$$\text{and } B = F/(1.0 + 2.0) = .3333 \text{ and } V = .6667$$

Since the proportions of F, B, and V_1 are fixed by fixing the value of V_s/B, the straight line BFV, representing the overall material balance must pass through F, the point B must lie somewhere on the line representing 60 mole-% heptane (specification line) and the point V_1 on the line A-A parallel to the heptane specification line located so that any straight line through F divides into segments such that $FV_1/FB = B/V_1$ as shown in Fig. 2. The location of the points B and V_1 on these lines and in other words, the determination of the composition of streams B and V_1 depends upon trial and error plate to plate calculations.

The trial and error plate to plate calculations are begun by assuming the location of the point B representing the composition of the liquid leaving the still. This assumption fixes the vapor V_1 leaving the stripping column. The calculation of compositions in the column can be started either at the top of the column or with the still.

If the calculations are started at the top of column, the liquid, L_1, leaving the top plate of the stripping column is located at the end of the equilibrium line through V_1. The stream B is the difference between the liquid feed and V_1 and is also the difference between the vapor and liquid passing each other between any two plates of the column.

Thus the vapor rising from the plate below the feed can be determined by a material balance and must lie on the line BL_1 so that $BV_2/BL_1 = F/V_1$. While the point V_2 can be located graphically, it is usually necessary to calculate the composition of V_2 by a material balance since the distances dealt with on the composition diagram are small and thus the graphical method is subject to considerable error unless unusual care is taken.

The liquid leaving plate 2 can now be calculated and lies on the equilibrium tie line from V_2. Again the bottom stream is the difference between the liquid L_2 and the vapor V_s and thus V_3 lies on the line BL_2 so that $BV_3/BL_2 = F/V_1$ or the point V_3 is preferably located by calculating the composition of the stream V_3 by a material balance.

The plate to plate calculation is continued until point B is passed. If the correct composition of the bottom product was assumed, smooth curves through both the liquid and vapor compositions will pass through the point B. Again it is usually necessary to estimate fractional equilibrium plate as in Fig. 2 where the number of equilibrium stages required to meet the specifications set in the problem are 1.8.

The estimation of fractional equilibrium stages in these calculations is not always entirely satisfactory since rigorously, the liquid compositions or the vapor composition does not necessarily form smooth curves. Where smooth curves are not formed, the quantities in the problem should be adjusted slightly in order to give an integral number of equilibrium stages.

It should be noted that while fixing the "boil-up" ratio and the heptane concentration of the bottom product with a given feed was sufficient to completely fix all of the variables of this problem, the composition of the top and bottom products is a function of reflux ratio. As the "boil-up" ratio was decreased from ∞ to 2.0, the mole-% of hexane in the bottom product containing 60.0 mole-% heptane increased from 26.0 to 30.0 mole-% and the number of equilibrium plates increased from .95 to 1.8. The "boil-up" ratio may be decreased until the number of plates required becomes infinitely large. Any further decrease in "boil-up" ratio represents an impossible operation.

A zone of infinite plates may develop in one of two

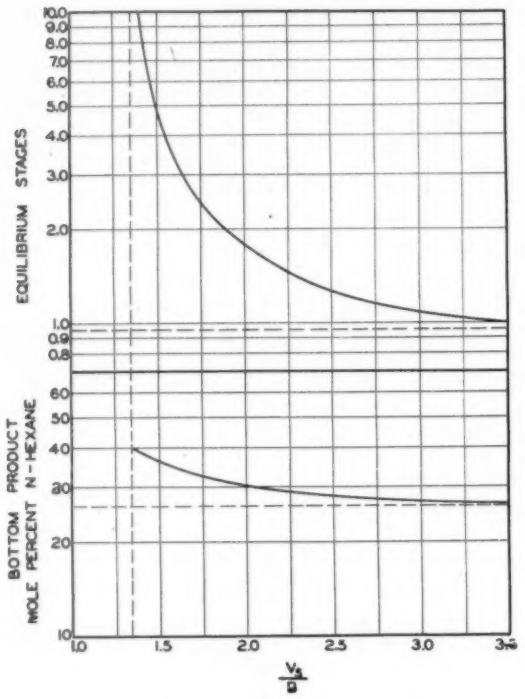

Fig. 4—Equilibrium stages and bottom composition as a function of "boil-up" ratio

ways as the "boil-up" ratio is decreased depending upon the feed composition and thermal condition, the bottom product specification, and upon the vapor-liquid equilibrium behavior of the particular system involved.

In one case, a zone of infinite plates develops along the heptane-hexane base in the middle of the column when the bottom product composition approaches that of a binary system before the overhead vapor approaches equilibrium with the feed liquid. In the second case, the zone of infinite plates develops at the top of the column (including the feed plate) when the overhead vapor approaches equilibrium with feed liquid before the bottom product approaches a binary composition. In the particular case under consideration the zone of infinite plates occurs when the bottom product, *B*, falls on the heptane-hexane base at 60 mole-% heptane. The operation is represented in Figs. 3a and 3b.

It has been pointed out that as the "boil-up" ratio is decreased, the composition of the bottom product increases in hexane content or the point *B* moves toward the hexane-heptane base of the composition diagram. When the point *B* reaches hexane-heptane base before the point V_1 on the straight line *BF* reaches the terminal point of the equilibrium tie line from the point *F*, a zone of infinite plates develops in the middle of the column and the composition of the liquid and vapor in the zone of infinite plates (or constant composition) consists of a binary system of the same components as those in the bottom product. In this case the equilibrium line through the feed intersects the base at the point *E* and does not intersect the bottom specification line within the diagram.

The calculation of the minimum "boil-up" ratio in this case becomes a somewhat arduous trial and error calculation which may be accomplished as follows:

Step 1. A minimum value of V_s/B is assumed.

Step 2. The top composition is calculated by a material balance based on the assumed value of V_s/B.

Step 3. The zone of infinite plates based on the assumption of Step 1, and a knowledge of the bottom product composition is determined by the usual methods of binary distillation.

Step 4. An arbitrary small amount of the missing component (in this case pentane) is added to the zone of con-

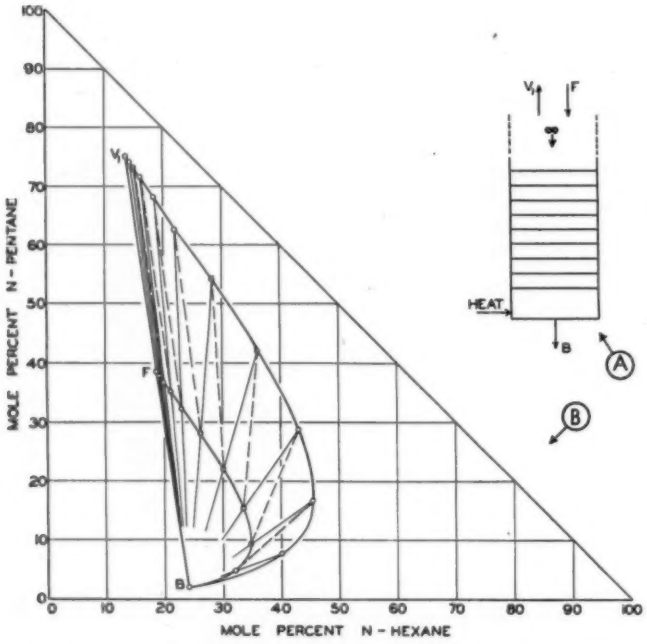

Fig. 5—(A)—Diagrammatic stripping column at minimum "boil-up" ratio. (B)—Composition diagram for a stripping column at minimum "boil-up" ratio

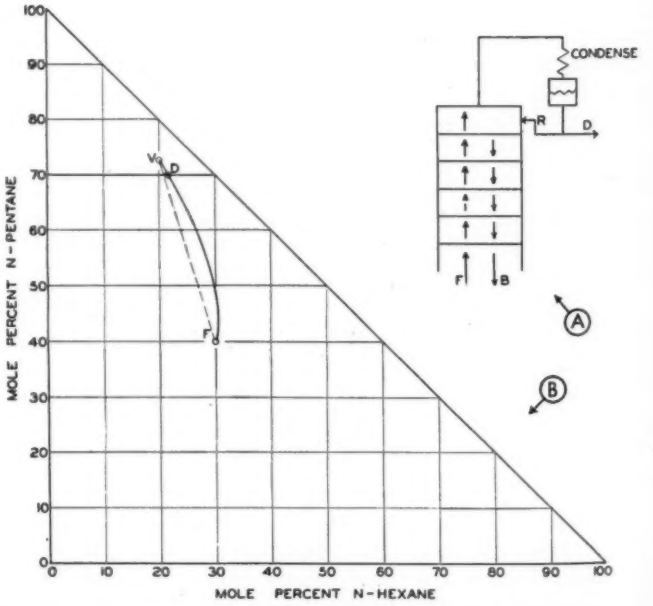

Fig. 6—(A)—Diagrammatic rectifying column. (B)—Composition diagram for a rectifying column at total reflux

stant composition and the plate to plate calculations are made from this composition toward the top of column. The amount of the arbitrary addition of the missing component is immaterial as long as it is small.

Step 5. When the correct value of V_s/B has been assumed it will be possible to check the values of the overhead product calculated from the overall material balance (Step 2) with the value calculated by plate to plate procedure from the zone of infinite plates.

In the special case of ternary distillation, a somewhat simpler procedure than that outlined under Step 4 is to calculate down to the zone of infinite plates from the top of the column after carrying out Steps 1 and 2. As shown in Fig. 3b the correct V_s/B can be bracketed rather easily by calculating one or two plates and plotting the path of vapor and liquid composition to determine whether it is possible to arrive at the hexane-heptane base at zone of constant composition.

This method is so sensitive that inaccuracies in reading K values or any other slight inaccuracies in calculation may change the path of the vapor and liquid compositions markedly, especially near the the heptane-hexane base. If the path of compositions is desired, the method outlined in the five steps can then be used with the V_s/B determined from the calculations from the top of the column. However, the method outlined in the five steps may be applied generally to all multicomponent distillation.

The calculation of the minimum "boil-up" ratio when producing a bottom containing 60 mole-% heptane from the aforementioned feed is summarized in Table 1 and illustrated in Figs. 3a and 3b. The final results of these calculations are a relationship between V_s/B and the number of plates summarized in Fig. 4 which also shows the variation in the amount of hexane in the bottom product.

In the second case, the zone of infinite plates develops when the bottom product, B, is so located that the point V_1 representing the overhead product falls upon the extension of the equilibrium tie line from the point F, or in other words, when the extrapolated line through the feed point intersects the bottom specification line within the diagram. In this case as the top of the column is approached by the plate to plate calculations from the bottom of the column; the fractionation accomplished per plate becomes smaller and smaller and becomes zero at the point V_1 and F as shown in Figs. 5a and 5b.

TABLE 1—Calculation from Top of Stripping Column for Assumed Minimum Reflux Ratio, V_s/B = 1.382 at 15 psia

Material Balance: $F = V + B = 1$
$1.382B + B = 1$
$B = 0.42$
$V = 0.58$

MATERIAL BALANCE

Component	Feed, moles F	Bottom Product, mole fraction x_B	Bottom Product, moles $= 0.42x_B$ B	$V=F-B$, moles	Distillate Product, mole fraction y_n
n-C5	0.4	..	....	0.400	0.6898
n-C6	0.3	0.4	0.168	0.132	0.2274
n-C7	0.3	0.6	0.252	0.048	0.0828
	1.0	1.0	0.420	0.580	1.0000

FIRST PLATE

Component	Distillate Product mole fraction y_D	K at 138°F and 15 psia	y_D/K	Corrected $y_D/K=L_1$, moles	$V_s=L_1-B$ moles
n-C5	0.6898	1.930	0.357	0.3562	0.3562
n-C6	0.2274	0.709	0.321	0.3204	0.1524
n-C7	0.0828	0.2555	0.324	0.3234	0.0714
	1.0000		1.002	1.0000	0.5800

SECOND PLATE

Component	V_2, mole fraction y_2	K146.5°F	y_{V2}/K	Corrected y_2/K: L_2, moles	$V_2=L_2-B$ moles	V_2, mole fraction y_2
n-C5	0.614	2.182	0.2810	0.280	0.280	0.483
n-C6	0.263	0.842	0.3192	0.318	0.150	0.2585
n-C7	0.123	0.305	0.4035	0.402	0.150	0.2585
			1.0037	1.000	0.580	1.0000

From Fig 3b, V_s/B is too high and the correct value is determined as 1.350 by calculations similar to those above. (See figure 3)

Calculation of compositions in zone of infinite plates

By material balance $L_\infty = V_\infty + B$

and $L_\infty x_\infty = V_\infty y_\infty + Bx_B$

For $V_s/B = 1.350$ and constant molal overflow,

$V = 1.350$, $B = 1.0$, and $L = 2.350$

and $2.350\ x_\infty = 1.350 y_\infty + x_B$

Since in the zone of infinite plates, the vapor and liquid are in equilibrium

$$y_\infty = Kx_\infty$$

and

$$2.350x_\infty = 1.350\ Kx_\infty + x_B$$

and thus,

$$\sum_{i=1}^{n} x_\infty = \sum_{i=1}^{n} \frac{x_B}{2.350 - 1.350\ K} = 1.0$$

COMPOSITION IN ZONE OF INFINITE PLATES

Component	Bottom Product mole fraction x_B	K 173.5°	2.350-1.350K	$\frac{x_B}{2.350\text{-}1.350K}$	Corrected $\frac{x_B}{2.350\text{-}1.350K}$
n-C6	0.40	1.271	0.634	0.631	0.636
n-C7	0.60	0.515	1.655	0.363	0.364
	1.00			0.994	1.000

CALCULATION OUT OF ZONE OF INFINITE PLATES BY ADDITION OF .002 MOLE FRACTION PENTANE

Component	L_n, mole fraction x_n	K 173.5°F	Kx_n	Corrected $Kx_n=V_n$, mole fraction or y_n	$1.350y_n=V_n$, moles	$V_n+B=L_{n-1}$ moles
n-C5	0.002	3.130	0.00625	0.00625	0.00844	0.00844
n-C6	0.6347	1.271	0.80600	0.80665	1.08880	1.48880
n-C7	0.3633	0.515	0.18700	0.18710	0.25276	0.85276
			0.99925	1.00000	1.35000	2.35000

PLATE, n-1

Component	L_{n-1}, mole fraction
n-C5	0.0036
n-C6	0.6334
n-C7	0.3630
	1.0000

The plate to plate calculations are carried out until the compositions reach those of the feed and vapor product.

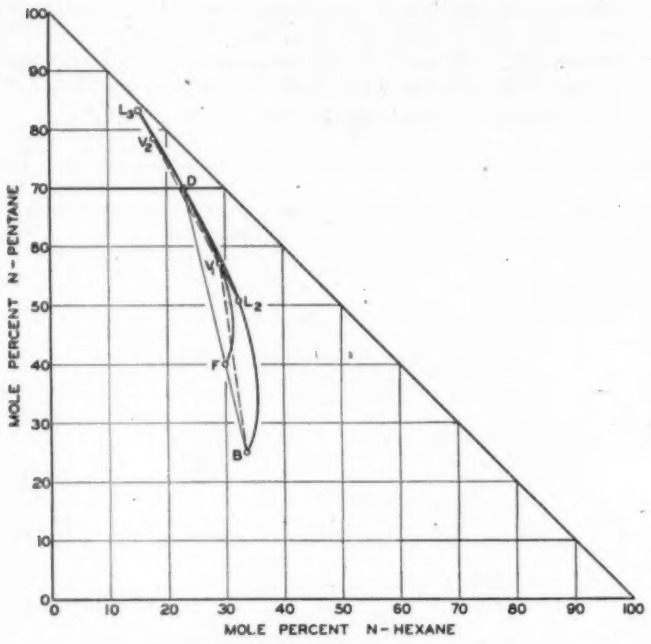

Fig. 7—Composition diagram for a rectifying column at finite reflux ratio

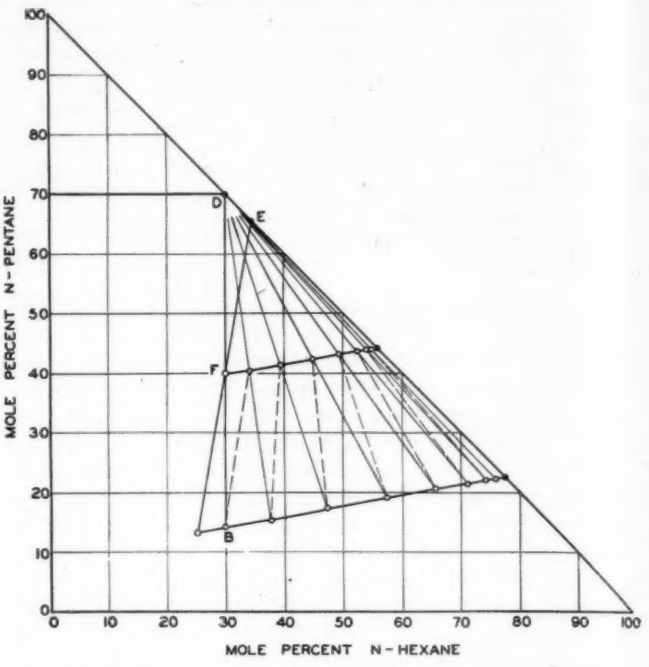

Fig. 8—Composition diagram for a rectifying column at minimum reflux ratio

As was demonstrated with binary systems, these curves show that the chief value of a stripping operation is the reduction of the concentration of the more volatile components in the bottom product. The overhead product must, of necessity, have a composition close to that of the feed composition since it leaves as a vapor from the plate to which the feed is introduced. Thus, the amount of the bottom product is small as compared to the amount of the overhead product and a stripping operation is not an efficient method of separating a mixture.

Rectifying Columns

The operation of rectifying columns using reflux has been described previously and illustrated by calculations with binary mixtures. The calculations required for the solution of the following problem are similar to those discussed in connection with "Stripping Columns."

A feed mixture containing 40 mole-% pentane, 30 mole-% hexane and 30 mole-% heptane is to be rectified to produce a distillate product containing 70 mole-% pentane. The feed is to be introduced as a saturated vapor (at its dew point) under the bottom plate of a rectifying column operated at one atmosphere pressure. Reflux is returned to the column at its bubble point temperature. It is desired to determine the relation between the "reflux ratio" and the number of equilibrium stages required. By "reflux ratio" is meant the ratio of the rate at which liquid is supplied to the top plate of the column to the rate at which the distillate product is withdrawn.

TABLE 2—Calculations on Rectifying Column for Minimum Reflux Ratio

The minimum reflux ratio (R/D) was determined as 1.190 by plate to plate calculations from the bottom of the column similar to the calculations for the stripping section.

Calculation of compositions in zone of infinite plates

By material balance:

$$V_\infty = D + L_\infty$$

and $$V_\infty y_\infty = Dx_D + L_\infty x_\infty$$

Since the vapor and liquid are in equilibrium in the zone:

$$y_\infty = Kx_\infty$$

From $R/D = 1.190$

$R = L = 1.190$, $D = 1$, and $V = 2.19$

and thus:

$$2.190 y_\infty = x_D + 1.190 \frac{y_\infty}{K}$$

or

$$\Sigma\, y_\infty = \Sigma \frac{x_D}{2.19 - \dfrac{1.19}{K}}$$

COMPOSITION IN ZONE OF INFINITE PLATES

Component	D, mole fraction	K, 139°F and 15 psia	$2.19 - \frac{1.19}{K}$	$\frac{x_D}{2.19 - \frac{1.19}{K}}$	corrected $= V_\infty$, mole fraction or y_∞
n-C5	0.7	1.960	1.583	0.442	0.443
n-C6	0.3	0.722	0.542	0.554	0.557
				0.996	1.000

CALCULATION OUT OF ZONE OF CONSTANT COMPOSITION BY ADDITION OF .005 MOLE FRACTION HEPTANE

Component	V_n, mole fraction y_n	K 139.5 °F	y_n/K	Corrected $y_n/K = L_n$, mole fraction or x_n	$1.19x_D = L_n$ moles	$L_n + D = V_{n+1}$ moles	$V_n + 1$, mole fraction = y_{n+1}
n-C5	0.441	1.975	0.2235	0.2227	0.2652	0.9652	.4408
n-C6	0.554	0.723	0.7610	0.7595	0.9024	1.2024	.5490
n-C7	0.005	0.265	0.0189	0.0188	0.0224	0.0224	.0102
	1.000		1.0034	1.0000	1.1900	2.1900	1.0000

The calculations are continued plate to plate until the feed and bottom product compositions are reached.

The rectifying column is illustrated in Fig. 6a and the composition diagram in Fig. 6b. The possible range of variation of reflux ratio is from $R/D = \infty$ corresponding to total reflux with no distillate product withdrawn and requiring a minimum number of plates, to some minimum value of R/D at which an infinite number of plates will be required.

As has been previously shown(2), at total reflux the difference between liquid and vapor passing one another between any two plates is zero and thus the quantity and composition of the bottom product B will be equal to the feed quantity and composition. Thus, the point F in Fig. 6b represents both the feed and bottom product.

The vapors leaving the bottom plate, V_1, will be in equilibrium with the bottom product, B, and therefore will be represented by the point V_1, in Fig. 6b at the end of the equilibrium tie line from B. As before, the point V_1 also represents the liquid from the plate above passing the vapor at total reflux. The vapor leaving the second plate from the bottom is represented by the point V_2 on the equilibrium tie line from the point V_1. The plate to plate calculation is continued in this manner until a vapor composition is reached which contains more than the specified concentration of pentane. The equilibrium stages are then estimated as before as .9 for this particular problem.

At a finite reflux ratio such as 2.0, the quantity of the distillate and bottom products is fixed by a material balance calculation as .3333 and .6667 mols per mol of feed since with saturated reflux (reflux at its bubble point), the quantity of bottom product is equal to the quantity of reflux returned to the column. Thus the line BFD representing the overall material balance must pass through the point F, the point D must lie on the specification line representing 70 mole-% pentane and the point B must be so located that the segments of the straight line BFD be in the ratio $R/D = DF/FB$, as shown in Fig. 7. As in the case of the stripping column, the compositions of the distillate and bottom products must be determined by a trial and error plate to plate calculation.

The composition of the distillate product, D, is assumed thus locating points B and D. The vapor leaving the bottom plate is located by an equilibrium calculation at the

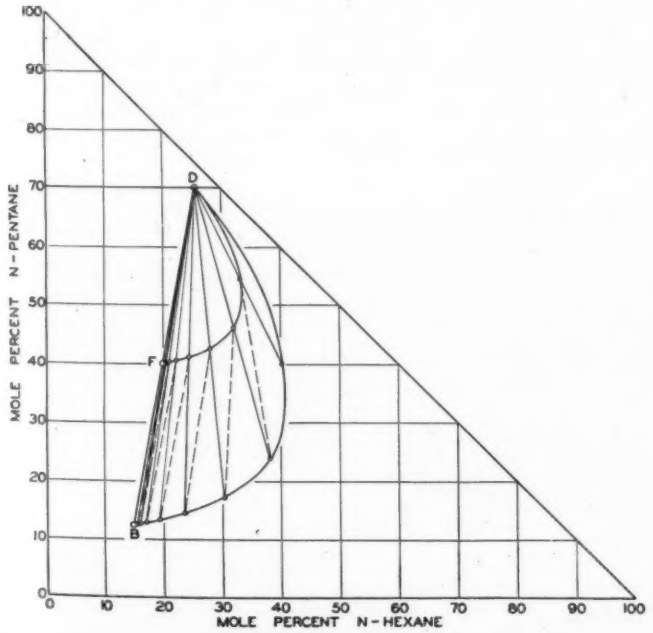

Fig. 10—Composition diagram for a rectifying column at minimum reflux ratio

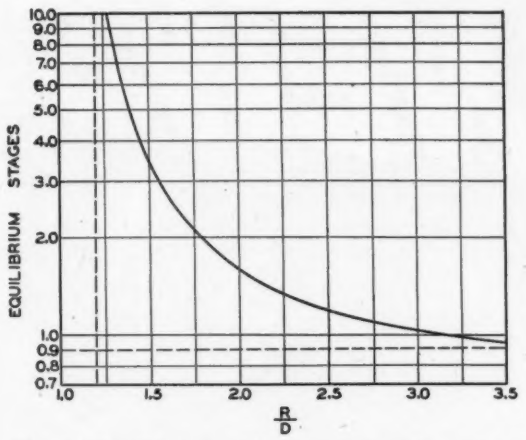

Fig. 9—Equilibrium plates as a function of reflux ratio

end of the tie line from the bottom product, B. The difference between the vapor, V_n, rising from the bottom plate and the liquid, L_{n+1}, going to the bottom plate is the distillate product and is located on the straight line, DV_n, such that $DV_n/V_nL_{n+1} = R/D$. Again it is usually preferable to calculate the composition of the liquid by a material balance because of the inaccuracies of the graphical procedure.

The plate to plate calculations are continued until the distillate product composition is passed. If the correct composition of distillate product, D, has been assumed, smooth curves through the liquid and vapor compositions will pass through the point D. As shown in Fig. 7 for the problem under discussion; when the reflux ratio is 2.0, 1.6 equilibrium stages will be required.

As the reflux ratio was decreased from ∞ to 2.0, the hexane concentration of the distillate increased from 21.5 to 23.0 mole-% and the equilibrium stages requirement increased from 0.9 to 1.6.

As in the stripping column, a zone of infinite plates may develop in one of two ways as the reflux ratio approaches the minimum depending upon the feed composition and thermal condition, the specification of the distillate product, and the vapor-liquid equilibrium data. In this case, the equilibrium tie line through the feed intersects the pentane-hexane base below the pentane specification at the point E. Thus as in the stripping column, the zone of infinite plates develops in the middle of the column and the composition of the liquid and vapor in the zone of constant composition or infinite plates consists of binary system of pentane and hexane, the two components in the distillate product.

The details of this calculation are so similar to those already discussed in detail when considering the stripping column, that the tabular summary of the calculations presented in Table 2 should be sufficient. These are also shown in Fig. 8. The relation between reflux ratio and equilibrium plates required for the problem are shown in Fig. 9.

If the feed composition is changed to 40 mole-% pentane, 20 mole-% hexane, 40 mole-% heptane with the same distillate requirements, at minimum reflux the zone of constant occurs at the bottom of the column since the equilibrium tie through the feed now intersects the distillate specification line within the ternary area at the point D. This is illustrated in Fig. 10.

References

(1) White, Robert R., Petroleum Processing, 1, 230 (1946).
(2) White, Robert R., and Boyd, W. T., Petroleum Processing, 1, 319 (1946).

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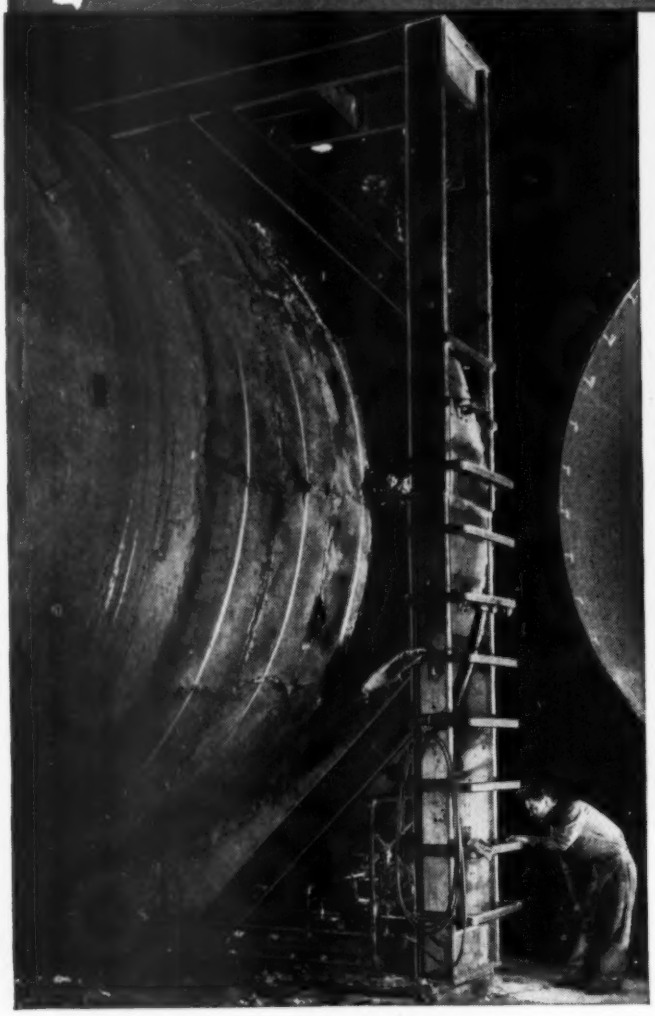
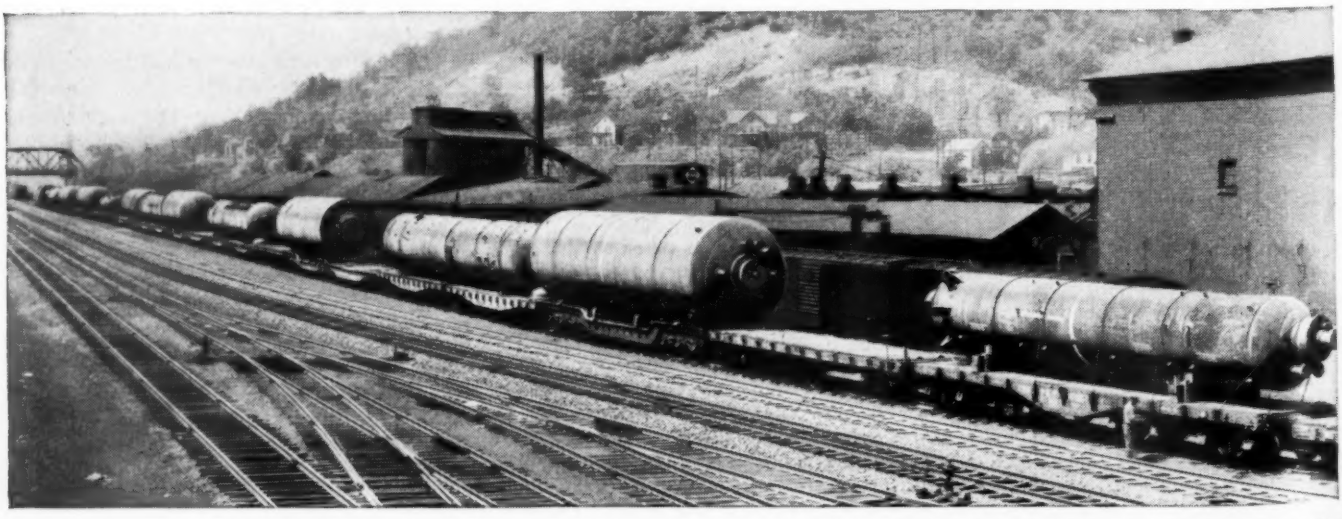


STRESS-ANNEALING BY THE INCH! (*above*) When a vessel is too long, or its diameter too great, to fit into one of the monster A.O. Smith furnaces, the engineers devise a portable furnace and stress-anneal section by section and the final closing seam.

MILLION ELECTRODE MAN. (*above*) In his twenty-one years of painstaking welding on SMITHway pressure vessels, John Binder has used up 975,744 SMITHway electrodes—at last count!

REFINERY ON WHEELS! (*below*) Not long ago this train pulled out of the A. O. Smith yards bearing almost the entire vessel equipment for a completely new refinery unit. The single shipment included coking drums, fractionating towers, stripping columns, flash chambers and coke chambers.



Patent Trends in Petroleum Refining

By PETER J. GAYLOR

Patent Attorney and Editor "The Technical Survey"

Developments disclosed in the patents reviewed below include:

Method for cracking hydrocarbons in the vapor phase to obtain high yields of aromatics and unsaturates

Increasing toxicty of petroleum oils used to impregnate wood

Use of metal hydride to dehydrate transformer cooling oils

New addition compounds from ethylene and propylene

Cracking to Aromatics and Gas

A METHOD for cracking hydrocarbons in the vapor phase so as to obtain high yields of aromatics and unsaturates is described in U. S. Patent 2,426,848 issued to Max B. Miller & Co.

In conventional non-catalytic cracking, when conversion to the desired yield is obtained, a hard tenacious crust of carbon is deposited on the walls, which involves trouble and expense. If conversion to the proper end point is not carried out, a by-product "pressure tar" is obtained which has a low value.

There has been proposed a method of cracking involving spraying of hydrocarbons upon a bed of coke heated from below. However, the bed soon becomes a dense mass, requiring shut down and removal of the packing material.

In the present patent, a process is disclosed wherein hydrocarbons are introduced into a deep incandescent bed of loose granular coke at a point spaced below the top of the bed so that the feed is forced to flow through a substantial portion of the bed. The desired temperature of the bed is maintained by burning coke at the bottom of the bed and depletion of the bed is prevented by coking liquid hydrocarbons at the top of the bed.

The process may be more readily understood by reference to Fig. 1. Charging stock from tank 4 is pumped into heater 7 and flash pot 8 and into inlet 25 of cracking vessel 20, and thence the oil is sprayed through orifices 27 onto the top of the incandescent bed of coke 14 where it is coked, cracked and vaporized.

Inlet tube 25 is provided with pulley drive 29 which rotates the inlet tube, on the bottom of which are attached rake fingers 31 which continuously rake the top coking section of the bed. Air is fed in at the bottom of the bed through inlets 21, in sufficient amounts to keep the coke incandescent; steam may also be introduced through pipes 22.

The gases and vapors taken off through pipe 40 are fractionated in tower 41. Liquid product is condensed out in tank 45, and gas is drawn off through outlet 46.

It is stated that this serves as a good means for producing styrene, benezene, toluene, ethylene, etc., although one obvious disadvantage is the dilution of the product with nitrogen from the air which would not only make separation of the gases difficult, but would also reduce condenser efficiency. Perhaps with the advent of cheap oxygen, the process may have commercial application.

Ash is drawn off the bottom 38 of cracking tower 20 and removed in dump carts 37. Temperature in the combustion zone is specified as 2800-3200° F., while that of the upper portion of the bed is kept at 1400-1800° F.

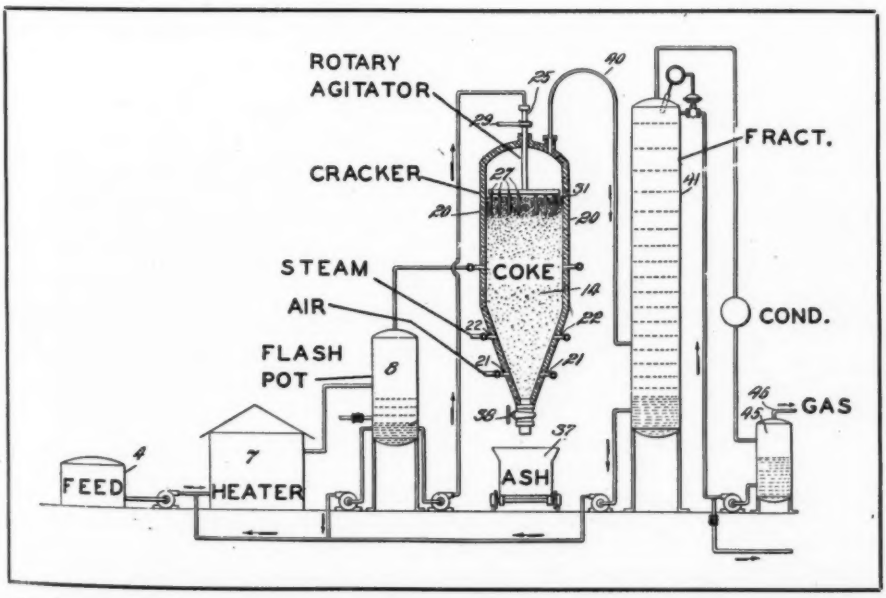


Fig. 1—Flow diagram of new method for cracking hydrocarbons in the vapor phase to obtain high yields of aromatics and unsaturates. U. S. 2,426,848

Wood Preserving Oils

THE petroleum industry is a source of a huge amount of aromatics, such as those employed for impregnating wood to prevent decay by fungus and insects. Petroleum aromatics apparently have not been as toxic to wood-destroying fungi as coal tar aromatics.

In U. S. Patent 2,419,212, the Southern Wood Preserving C. discloses a process which is claimed to increase considerably the toxicity of petroleum oils. In oily wood impregnants, it has been found that fractions boiling below 315° C. have a relatively high toxicity, whereas those boiling in the 315°-355° C. range have a relatively low toxicity. Besides, when mixed together, the toxicity of the blend is much lower than calculated due to some sort of blanketing effect of the higher boiling fraction on the lower one. It has been found that if there are provided 70% of materials of ring structure content in the 210°-355° C. range, and certain specific gravity limits are set on the 315°-355° C. fraction, the toxicity blanketing effect of the 315°-355° C. fraction can be limited to an extraordinary degree.

If petroleum aromatic fractions are taken as starting material and subjected to high pressure cracking or

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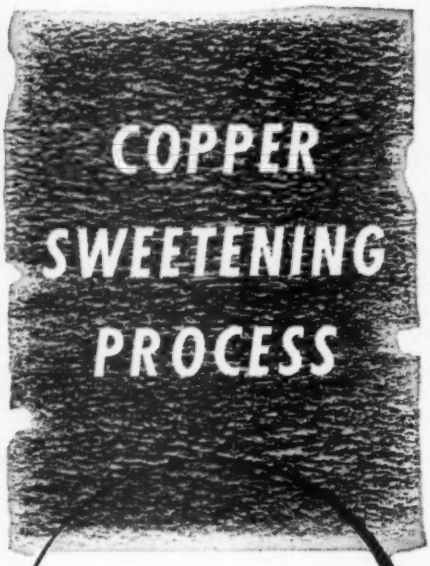

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reforming, a reformed oil can be produced having at least 70% aromatics in the 210°-355° C. range which would also have a specific gravity of 1.1175-1.3888. The toxicity blanketing effect of the 315°-355° C. fraction of this oil is reduced to an unobjectional point.

Transformer Oils

IT IS important that oils or other organic liquids, used as cooling agents in transformers, be free from substantially all water. Even as much as 0.1% moisture in the oil generally renders it unfit for use, and the maximum allowable moisture in a high voltage transformer is 0.04%. The decrease in dielectric strength of a transformer oil is very rapid with increase in moisture content of the oil, particularly when passing from a dry oil to one with a moisture content of one part in ten thousand.

To remove this water, various methods have been practiced, involving blotting presses, sodium treatment, calcium chloride treatment, etc. In the case of sodium treatment, the reaction is not only dangerous, but the reaction also generates heat which tends to break down the oil.

U. S. Patent 2,399,192 issued to Metal Hydrides, Inc. of Beverly, Mass., discloses a new method for dehydrating transformer oils, utilizing a metal hydride such as calcium hydride. The hydride reacts with the moisture to produce the metal oxide and hydrogen. One method of treatment involves forcing the oil slowly through a bed of the hydride. Another includes distilling the oil in a vacuum and passing the vapors through a bed of finely divided calcium hydride.

Chemicals

SOME interesting crystalline addition compounds have been obtained by Solvay Process Co. by reacting various olefins with *nitrosyl chloride* (NOCl). For example, U. S. patent 2,417,675 discloses that propylene and ethylene can react in a manner as to form colorless crystalline bis-nitroso chlorides. The ethylene derivative melts at 71-2° C., while the propylene addition product melts at 91-2° C. Due to the reactivity of the chlorine atom and of the adjacent nitroso group, these new products may be employed in a number of syntheses.

Such products may be obtained by reacting propylene or ethylene with nitrosyl chloride at a temperature of −25° to 60° C., in liquid phase, in presence of a volatile saturated halogenated hydrocarbon as solvent, and crystallizing the addition product from the mixture. The presence of a catalyst such as nickel or a heavy metal chloride (nickel chloride) gives an increased yield of the addition compound. Optimum temperature is about 20°-40° C.

One patent to Union Oil Co. of California (U. S. 2,420,938) describes the use of *nitric acid as a selective solvent for extraction of organic nitro compounds,* such as nitrocyclohexane, nitroisooctane, etc. Nitric acid of 70% strength at 20° C. has been used to separate nitrocyclohexane from nitroisooctane, the latter compound being found in the upper phase.

Nitro 3-methyl pentane was separated from 3-methyl pentane by extraction with 90% nitric acid at about −5° C., the hydrocarbon being in the upper phase.

Selected Patents of the Month

U. S. 2,425,512 and 33 (Texas)—Thickness measurement.
U. S. 2,425,535 (Standard Oil Development)—Cocoanut charcoal for isomerization.
U. S. 2,425,572 (Standard Oil Development)—Alkylation.
U. S. 2,425,671 (Socony-Vacuum Oil Co.)—Preparing drying oil with polymerization catalyst.
U. S. 2,427,212 (Pure Oil)—Removal of peroxides from oils with phenols.
U. S. 2,427,326 (Socony Vacuum Oil Co.) — Breaking water-in-oil emulsions.
U. S. 2,427,341 (Universal Oil Products) — Fluid catalytic converter.
U. S. 2,427,501 (Sinclair Refining)—Rust inhibiting oil.
U. S. 2,427,589 (Atlantic Refining)—Refining oils with sludge-forming agent.
U. S. 2,427,640 (Shell Development)—Polymerizable diester of sulfonyl diglycolic acid.
Can. 443,336 (Texaco Development)—Detergentized Diesel lubricant.
Can. 443,675 (Shell Development)—Separating butane and butyl sulfate.
Can. 443,873 (Shell Development)—Converting allyl amine into acrylonitrile.
Can. 443,875 (Standard Oil Development — Foam suppressor for soluble oils.
Brit. 590,980 (Shell Development)—Recovery of hydroarmomatic cycloparaffin hydrocarbons.
Brit. 591,101 (Standard Oil Development) — Supercharged aviation engine operation.
Brit. 591,358 (Standard Oil Development) — Alkyl halide separation.
Brit. 591,406 (Girdler Corporation)—Alkylation.
Brit. 591,373 (Shell Development)—Catalytic cracking.
Brit. Appl. 22,179/47 (Anglo-Iranian Oil) — Catalytic cracking.
Brit. Appl. 21,728-9/47 (California Research Corporation)—Dicarboxylic acid anhydrides.
Brit. Appl. 21,283/47 (Shell Development) — Diketones.
Brit. Appl. 21,285/47 (Shell Development) — Unsaturated ketone stabilization.
Brit. Appl. 21,865 & 978/47 (Standard Oil Co.) —Synthesizing hydrocarbons.
Brit. Appl. 20,888-9/47 (Socony)—Desulfurizing oils.
Brit. Appl. 21,063-7/47 (Shell Development)—Solvent extraction.
Brit. Appl. 20,435/47 (Socony Vacuum)—Aminoalkyl derivatives.
Brit. Appl. 20,848-9/47 (Socony Vacuum) — Gelling process.
Brit. Appl. 20,887-9/47 (Socony Vacuum) — Treating oils.
Brit. Appl. 21,014/47 (Standard Oil Development)—Hydrocarbon drying oils.
Brit. Appl. 1108-10/47 (Houdry Process) — Catalyst.
Brit. Appl. 18,007/47 (Standard Oil Development)—Detergents, etc.
Brit. Appl. 2104/47 (Texaco Development)—Alkylated thiophene compounds.
Austral. Appl. 12,883-4/47 (Shell Development) —Combating noxious organisms.
Austral. Appl. 13,414/47 (Anglo-Iranian Oil)—Detergents.

will this NEW oil additive open NEW profit opportunities for you?

Want to get ahead of competition in the race to meet the growing number of motor oil specifications that call for a high viscosity index?

Want to turn your low-viscosity index base oil stocks into top-quality lubricants—without the expense of extreme refining?

Want to meet the viscosity requirements of both the "W" and the usual SAE grades with one lubricant—thereby reducing both distribution and manufacturing costs?

Those are some of the profit opportunities opened up by Santodex, a new oil additive developed by Monsanto to improve viscosity index.

Depending on the base oil stock with which it is blended, Santodex makes possible viscosity indices up to 140 or higher—often at a significant saving over other viscosity-index-improvers previously available.

To many lubricant manufacturers this will mean important production and distribution economies like those listed above. It will also mean that now you can produce—at a profit—motor oils with all the benefits to a motorist that go with a high viscosity index:

—easier starting in zero weather.
—lower oil consumption at high temperatures.
—better oil-film protection for engine working parts.

An all-hydrocarbon material, Santodex is readily soluble in mineral oil and does not stratify or separate from a lubricant. At normal concentrations, it has no significant effect on the gravity, flash or fire points, pour point, neutralization number, carbon residue or color of the base stock with which it is blended. It can be readily compounded in conventional refinery equipment.

For full details and liberal samples of Santodex, wire, phone or mail the coupon below. MONSANTO CHEMICAL COMPANY, Petroleum Chemicals Department, 1700 South Second Street, St. Louis 4, Missouri. On the West Coast contact District Sales Offices in Los Angeles, San Francisco, Seattle. In Canada: Monsanto (Canada) Limited, Montreal.

Santodex—Physical Properties

Specific Gravity	0.899
A.P.I. Gravity	25.9
Pounds per gallon	7.49
Flash Point (C.O.C.)	350° F.
Pour Point	25° F.
SUS Viscosity	
a. 100° F.	35,500
b. 210° F.	2,160
Neutralization Number	.03
Color, ASTM	2½
Ash	.01%
Metals	None

How Santodex Improves Viscosity Index of Several Typical Oils

Per Cent Santodex	0%	1.0%	2.0%	4.0%	8.0%
Pennsylvania	105	120	129	136	142
Solvent Mid-Continent	95	109	117	129	140
Conventional Mid-Continent	78	93	105	115	128
Coastal	25	63	82	101	118

MONSANTO
CHEMICALS AND PLASTICS

SERVING INDUSTRY . . . WHICH SERVES MANKIND

MONSANTO CHEMICAL COMPANY
Petroleum Chemicals Department
1715 South Second Street, St. Louis 4, Missouri

Please send ☐ technical data and ☐ a liberal sample of Santodex to test with our base stocks.

Name.....
Title.....
Firm.....
Street.....
City.....State.....

Double Float Rotameter Provides Continuous Viscosity Measurement

BY USING two separate floats in the same tube, a rotameter type of instrument measures viscosity of a fluid in motion, for example through a pipe.

One of the two floats is unaffected by variations in viscosity and its position is determined solely by the rate-of-flow. The other float is sensitive to variations in both viscosity and rate-of-flow. If the flow is kept at a constant rate, the visible effects on the position of the second float will therefore be by viscosity variations. Hence viscosity can be read directly by the difference in positions of the two floats.

One of the several modifications in design is shown in Fig. 1. Tube 20 is a slightly tapering cylinder with a constant diameter between the lower end and point 43, then widening up to the top end. It includes three slender beads 45 on its inner wall for guiding the floats in reasonably central position with little friction.

Fluid enters port 28 passes upward through tube 20, and out port 30, as shown by the arrows. Float 47, connected by rod 46 to weight 49, is not affected by variations in viscosity because of its design. It is in the form of an inverted cup, or truncated cone.

Float 51 is a simple cylinder riding in the fluid motion above float 47. Its design renders it sensitive to both flow-rate and viscosity effects.

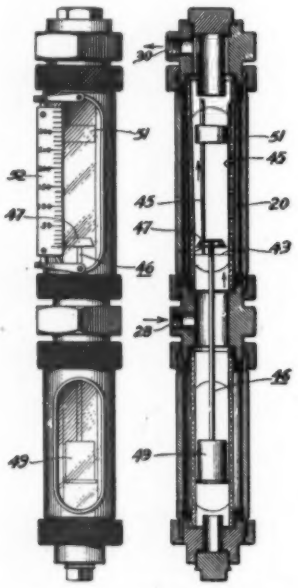


Fig. 1—When float 47 indicates constant flow rate of fluid through instrument, float 51 will show the viscosity (U. S. 2,426,393)

In operation, fluid is sent through the instrument at a constant flow-rate. Then a calibrated scale, 52, fastened vertically along the outside of the sight glass on tube 20, is set with its zero point at the level of float 47. The position of float 51 indicates the viscosity directly.

U. S. 2,426,393, issued Aug. 26, 1947, to Kermit Fischer, assignor to Fischer & Porter Co.

Obtain Nearly Pure Hydrocarbon With Separator for Paraffin Wax

SEPARATION OF the components of homogeneous mixtures of paraffin waxes into close-cut fractions of nearly pure hydrocarbons can be accomplished by a new method and apparatus which uses a solvent and stratification along with recrystallization.

The equipment is shown schematically in Fig. 2. A tall, vertical, cylinder 1 is packed with a filter medium such as sand. Tube 1 is surrounded by a concentric jacket 3. In the annular space 3, a movable heating element 5 floats on the surface of liquid coolant 6.

Coolant is pumped by pump 27 from a reservoir 7 through conduit 8 to a long manifold of valves 9, which are manually controlled so that only one admits coolant to the jacket at a time and at a point just below the element 5.

The coolant is circulated by pump 12 through heat exchanger 13, packed with dry ice. Valve 10 is provided to cut off the supply of coolant after the jacket 3 is first filled. Valve 16 bleeds off the coolant so as to lower the element 5. Solvent is circulated through the system by pump 19.

In one application, pentane was used to separate a refined paraffin wax of 135° F. melting point, trichloroethylene being the coolant. The unit was started by filling column 1 to the level of the sand with pentane and the jacket with coolant.

A quantity of wax was introduced above the sand, and the heater was turned on to give the sand in the neighborhood of the heater a temperature of about 20° F., the trichloroethylene being maintained at about — 60° F. Solvent circulation was started and coolant was allowed to bleed off slowly.

Wax dissolved in the pentane and began to pass downward in solution. It was in turn precipitated on the

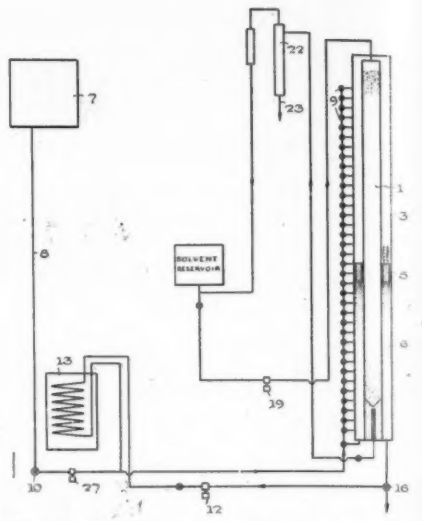


Fig. 2—Schematic layout of separator for close fractionation of wax (U. S. 2,427,042)

sand as it flowed into the cooler region. As the temperature was raised in that region by the lowering of element 5, solid wax redissolved, and again precipitated the cycle repeating on through the apparatus.

Each time the wax was deposited, however, it was deposited non-uniformly depending upon the solubility-temperature relationship in the solvent for each of the wax components. The more soluble components were deposited lower in the column—at the lower temperatures, the separation becoming more and more distinct as the process continued.

When the solvent coming out at the bottom of the column contained some portions of wax, the solution was passed to stripper 22, where the solvent was recovered and the components taken off in succession at 23. A total of 28 fractions, ranging in %-wt. between 2 and 5 and in melting point from 50° F. to 146.3° F., were recovered from the 135° F. m.p. wax.

U. S. 2,427,042, issued September 9, 1947, to John R. Bowman, assignor to Gulf Research & Development Co.

Automatic Locator Is Developed For Slide Rule Decimal Points

A TINY RATCHET wheel and pawl mechanism has been adapted for use in the design of an automatic decimal point locator for slide rules.

The decimal point is indicated on a dial which is automatically operated in response to movements of both the slider and the rider of the rule in the course of calculations. The device is mounted directly on the rider. A modified form has been developed also for use on a rotary type slide rule.

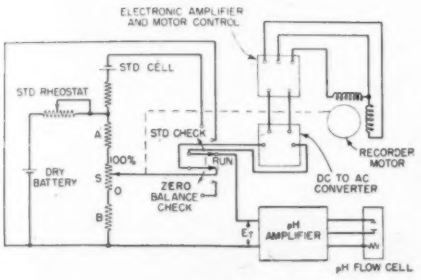
U. S. 2,427,171, issued September 2, 1947, to Arthur F. Eckel, assignor to Myrtle Scott Eckel and Louise Wickersham Pickett, Chicago.

What's New!

Edited by
WILLIAM C. UHL
Equipment Editor

EQUIPMENT . . . MATERIALS . . . PROCESSES . . . LITERATURE

1—pH Recorder



The new Bailey pH Recorder is of the electronic type, operating without a galvanometer. It is said to balance automatically and continuously the potential furnished by the electrodes and electronic amplifier against a known reference potential. The continuous balance of the recorder is illustrated in the circuit diagram shown. Various combinations are available. Two records of pH on a single chart may be obtained in different colored inks. Automatic pneumatic control is also available and two controllers may be installed in most recorders. Standard types of pH electrodes are furnished —flow or immersion type. Complete instrument consists of a Beckman Electrode Assembly and Amplifier and a Bailey Potentiometer Recorder. Bailey Meter Co.

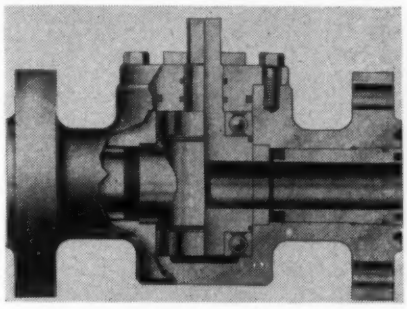
2—Viscometer

Rapid measurement of the kinetic viscosity of extremely viscous oils or similar materials is possible with the new Gardner Visco-Meter. It is said to be especially adapted to butadiene polymers, transparent oils, chlorinated rubber solutions, and the like. The instrument consists of a precision bore Pyrex glass sample tube engraved with 10 equidistant marks. The tube is clipped to a metal frame and arranged so that it can be revolved ½ revolution with a rack and pinion. It is used in a constant temperature water bath. In operation, the time is recorded for the bubble of air in the oil in the tube to travel between two marks. The tube is calibrated so that the time in seconds is equal to the viscosity in stokes. The entire length of the tube between marks is used for most liquids. In the case of extremely viscous materials, the distance between any two marks can be used and viscosity calculated quickly on a proportional basis. Henry A. Gardner Laboratory, Inc.

For More Information

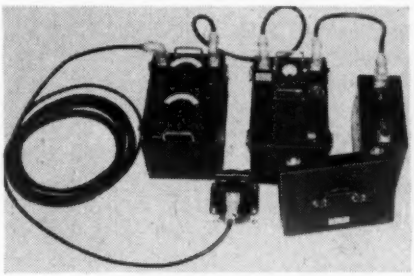
Use one of the attached business reply cards, which requires no postage, to request additional details or literature on any items reviewed in "What's New!" Just circle the numbers corresponding to the numbers on the items you're interested in, fill in the bottom of the card, and drop it in the mail.

3—Non-lubricated Plug Valve



The "LJS Plug Valve," for quick-opening, high pressure, fluid control, features a straight, non-tapering plug that requires no lubrication and is suspended top and bottom on sealed ball bearings. Very little torque is needed for the quarter turn to open or close the valve at high pressures and temperatures, the manufacturer states. Plug does not touch body at any point, keeping friction to a minimum. Expansion glands behind each seat have just enough "give" to prevent seizure or sticking. Glands are held in place by replaceable steel retainer sleeves. Fluid does not come into contact with valve body. All wearable parts —plug, seats, glands, and sleeves— can be replaced on the job without special tools, it is said. Flange and screw type LJS Plug Valves are available in ferrous, non-ferrous, and special metals for refining, processing, pipe line, or oil field service. Johnson Foundry and Machine Co.

4—Thickness Gage



Non-destructive inspection and testing of metallic containers, vessels, and piping while in service is claimed for the new Scott "Electroflux" Thickness Gage. Measurement is made from one side and is accurate to within ±3%, it is said. Range of instrument is from 0.125 to 2.25 in. of steel thickness. In operating form, it combines a four-electrode probe, a small six-volt battery, an electronic voltmeter and power supply for measuring the low voltage output from the probe, and inter-connecting cables to permit easy movement of the probe at a distance from the metering equipment. Portable, it weighs 55 lbs. Scott Electroflux Co.

5—Cutting Oil Additives

Two new cutting oil additives—"Cresol Z-2" and "Cresol Z-2A"—are reported to be adapted to the machining of SAE steels, Monel metal, chrome, chrome-molybdenum, chrome-nickel, tungsten, and the stainless steels. They are modified sulfur chlorides, containing as high as 40% sulfur and 30% chlorine, and are miscible in all proportions in petroleum fractions. Manufacturer states they will not cause, but rather prevent, skin infections. Lubricants, Inc.

More "What's New!" Items, Page 875

Huge new lube plants will operate on FILTROL

Year after year more petroleum refiners have depended on Filtrol Adsorbents for premium quality refining with greater flexibility, lower labor costs, higher efficiency, and minimum plant investment. This is the reason the projected, great new lube plants now in process of construction, decided on Filtrol contacting or Filtrol fractionation for finishing lube stocks—a tribute to unquestioned leadership in the field of petroleum adsorbents.

FILTROL CORPORATION

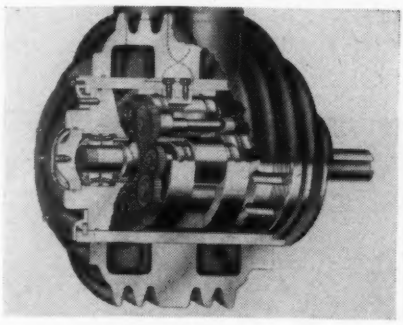
General Offices: 634 South Spring Street, Los Angeles 14, California
Plants: Vernon, California, and Jackson, Mississippi

PRODUCT OF **Filtrol*** **ADSORBENTS AND CATALYSTS**

RESEARCH AND DEVELOPMENT

* FILTROL: Trade-Mark Filtrol Corporation, Reg. U.S. Pat. Off.

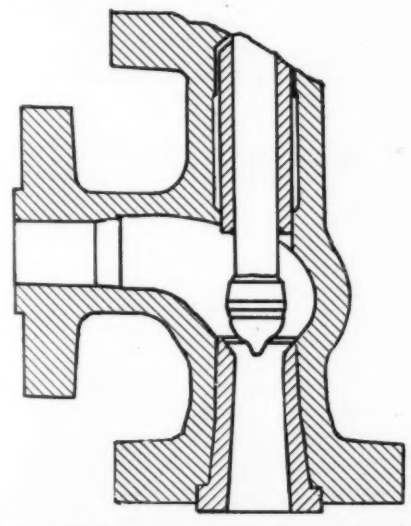
6—Rotary Pump



Said to have specific application in the petroleum and process industries because of its anti-friction bearing design, the new "Rotocycle" positive displacement pump is particularly suited to handling non-lubricating liquids. It is of the fixed volume, double-acting type, with all operating parts held in hydraulic and dynamic balance. It features a fixed, closely held clearance between the circumference of the four half-moon shaped impeller vanes, the sidewall of the pump chamber, and the rotor. Operation is quiet and without pulsation, and in either direction. A volumetric efficiency of 90-95% is claimed, with overall operating efficiencies of 85-90%. Rockwell Mfg. Co., Hydraulics Division.

7—High Pressure Valve

"Venturiflo" Diaphragm Control Valves are designed primarily for high pressure applications such as cracking still operation under severe coking conditions, synthesis of gaseous compounds, high pressure steam reduction, and the like. The new line of valves can handle many jobs on the current fluid catalytic crackers as well as those in the new Fischer-Tropsch process plants, it is said. The seat is designed as a section of a Venturi throat, insuring minimum turbulence, and is lapped into the valve body, eliminating gaskets. Valve body and bonnet is provided with flushing connections for use in coking service in petroleum cracking. Available in sizes from ½ through 1 in. with screwed ends, and 1 through 8 in. in flanged ends. Valves can be provided for 150, 300, 600, 900, and 1500 lbs. ASA ratings. Valve bodies available in cast iron, bronze, cast steel, and Types 304 and 316 stainless. Valves of this type are being supplied on special applications under pressures as high as 30,000 psi. and temperatures between —225 and +1000° F. Hammel-Dahl Co.

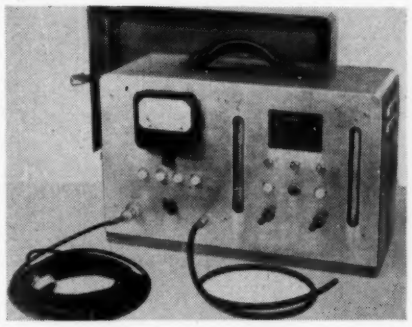


8—Pneumatic Hammer

The new J-10 Utility Jackhamer is said to have a wide variety of applications in maintenance work, and because of its light weight is suitable for overhead work on a ladder or scaffold. It can be used for star drilling, light paving breaking, chiseling, or channeling, by means of adapters. Includes a built-in oil reservoir in the handle for lubrication. Throttled control permits the selection of the right amount of force for each job and each tool. Ingersoll-Rand Co.

9—Toxicity Analyzer

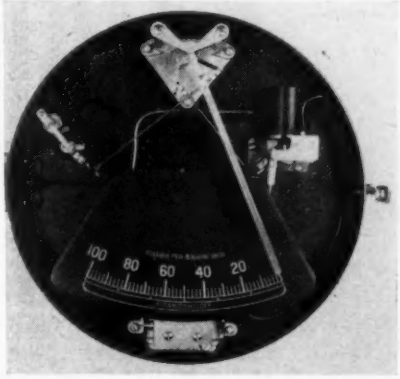
Working on the principle of electrical conductivity of solutions, the new Micro-Gas Analyzer determines the concentration of gases or vapors in their toxic range. Any gas which will ionize in water, with or without heat, can be analyzed. Included are



all chlorinated hydrocarbons, carbon disulfide, hydrogen sulfide, and sulfur dioxide. A small pump draws gas through a flowrator and into a combustion furnace, where it is decomposed. It then passes into a cell through which a solution is being pumped. As the decomposed gas dissolves, the electrical conductivity changes are measured on a meter calibrated in parts/million of the gas being analyzed. Included with the instrument is a chart which indicates flow rate of both water and air for the gas being tested. The unit is portable, weighing about 33 lbs., and operates from 110 AC, 60 cycle current. Davis Emergency Equipment Co., Inc.

10—Pneumatic Transmitter

Transmitting and receiving mechanisms are exactly matched and perfectly linear in calibration in the new Foxboro Model 42 Pneumatic Transmitter. This is claimed to result in improved accuracy and simplified calibration. Available with either concentric or eccentric scale, the transmitter may be used for any process measurements such as flow, pressure, level, humidity, or temperature. A 12-inch round case, larger than previous models, provides room for additional units, such as alarm or signal devices, if desired. Foxboro Co.



More "What's New!" Items, Page 877

His laboratory is the only full-scale aviation engine test cell in the petroleum industry.

This man is working for you!

It provides him with performance data on additive-improved aviation fuels and lubricants . . . under simulated service conditions.

. . . checking additive-improved aviation products.

This is but one example of how the work of over 2000 research scientists and technicians . . . backed by the facilities of the largest petroleum laboratories in the U. S. . . . can aid you when you use PARAMINS.

Let PARAMINS help improve your products and increase your sales!

PARAMINS* make good motor oils and fuels better!

ADDITIVES WITH A BACKGROUND

PARAMINS ADDITIVES ARE KNOWN BY THE BRANDS:

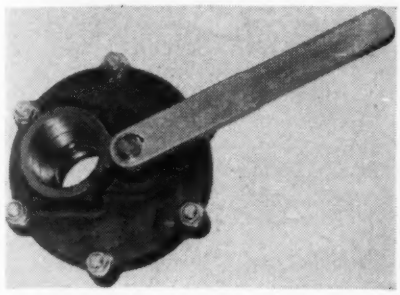
PARATONE —*for improved viscosity index.*
PARAFLOW —*for lower stable pour.*
PARATAC —*for tacky oils and greases.*
PARAPOID —*for E.P. gear oils.*
PARANOX —*for inhibiting corrosion and oxidation.*
PARASHEEN—*for better appearance.*
PARADYNE —*for improved gasoline.*

*Trade Mark

ENJAY COMPANY, INC.

15 WEST 51st STREET, NEW YORK 19, N. Y. • AGENTS AND DISTRIBUTORS THROUGHOUT THE WORLD

11—Quick-opening Valve



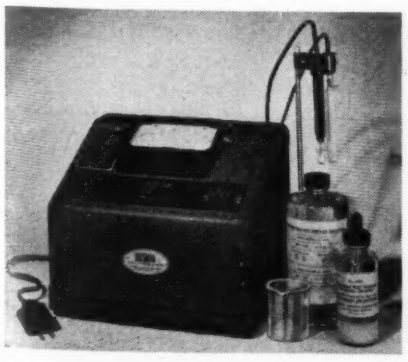
Designed for butane and similar service, a new quick-opening, heavy-duty steel valve employs a patented "Shear-Seal" principle which is said to provide metal-to-metal seal that is self-aligning and automatically compensates for wear. The valve illustrated fits 2-in. pipe with a straight-through opening of 1-11/16 in. diameter. Force required at end of handle for operation at 200 psi. is 18 lbs. The valve is also available for water or corrosive service with a heavy cast bronze body. Saval Co.

12—Ratio Controller

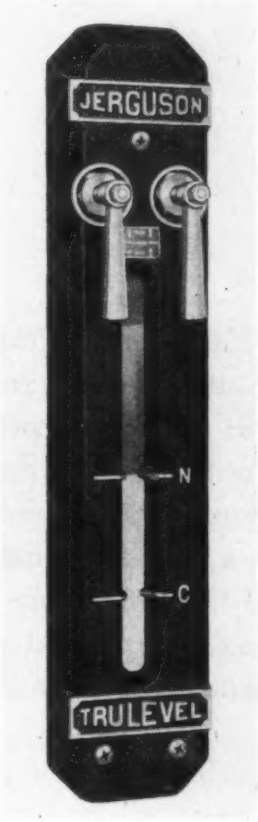
A new air-fuel ratio controller for use in large industrial furnaces employs electric-motored valve drives—needs no hydraulic piping. A balance-type instrument, it can be adjusted manually for operation with fuels of various Btu. content. As fuel flow changes, it maintains the air-fuel ratio constant at the desired control point; or, it can provide automatic variation of ratio. Leeds & Northrup Co.

13—Portable pH Meter

The Model H, Beckman, portable, glass electrode, pH Meter features a built-in temperature compensator dial calibrated to 0 to 100° C, which can be set at the temperature of the test sample. This automatically adjusts the measuring current to compensate for the effect of temperature on the EMF of the glass electrode, eliminating the need for a pH-temperature correction chart. Changes in circuit constants due to temperature effects are approximately 0.1 pH unit in the first hour and negligible thereafter, it is claimed. The unit covers the full scale from 0 to 14 pH and can also be set for making millivolt readings from 0 to ±410 mv. Accuracy is 0.03 pH or 2 mv. Net weight is 14 lbs., measurements are 12 x 9½ x 8½ in. overall. It can be plugged into any standard 110 v. 50/60 cycle AC line. Power consumption is 35 watts. Instrument is supplied with electrode support rod and holder, glass and Calomel electrodes, pint of pH 7.00 concentrated buffer, 100 ml. saturated KCl solution, and a 50 ml. Pyrex beaker. National Technical Laboratories.



14—Level Gage



The Jerguson "Trulevel" Gage can be read and quickly checked at a remote reading point. A fully steam jacketed datum column located at the boiler drum duplicates the temperature and specific gravity conditions existing in the drum. The patented checking feature involves a quick operation of turning the two valve handles on the cover of the gage. Jerguson Valve & Gage Co.

15—Pipe Clamp

A commercial pipe clamp made of chrome vanadium steel as a one-piece drop forging and equipped with a U-bolt of the same material has been developed for high temperature service. It is expected to meet the major portion of such requirements at temperatures up to 1000° F. without a high first-cost penalty. The clamp is available in seven sizes from 4 to 16 in. In load rating tests, a 14 in. clamp reached a tensile load of 66,300 lbs. before destruction. Grinnell Co., Inc.

Trade Literature

16—Hydrogen

Hydrogen, 32 pages of descriptive information on the various modern processes for manufacturing hydrogen — Girdler's hydrocarbon-steam, the water gas method, steam-iron, methanol-steam, electrolytic, and others. Includes useful data for problems involving hydrogen. Girdler Corp., Gas Processes Division.

17—Stuffingboxless Pump

Stuffingboxless Pumps, a reprint of a technical paper under the same title delivered at the Pacific Chemical Exposition, October 21-25, at San Francisco, and describing a newly developed centrifugal pump for oil refineries, pipeline systems, and pumping stations which uses a mercury and mechanical seal instead of costly-maintained stuffingbox. Byron-Jackson Co., Pump Division.

18—Continuous Viscosimeter

Viscorator, Catalog Section 88-C, a four-page folder giving details and engineering data on the "Viscorator", a continuous viscosimeter for measurement, recording, and control of gas or liquid viscosities from 0.0008 to 800,000 centipoises. Fischer & Porter Co. (A patent recently issued to this company on a new design for a viscosimeter is reviewed in this month's patent section——see page 872).

19—Stainless Castings

A Quarter Century of Pioneering in Stainless Steel Castings, a 44-page booklet describing the progress of technology in the manufacture of stainless steel castings since 1922. Includes a table of analyses, specifications, and applications for various alloys. Cooper Alloy Foundry Co.

More "What's New!" Items, Page 879

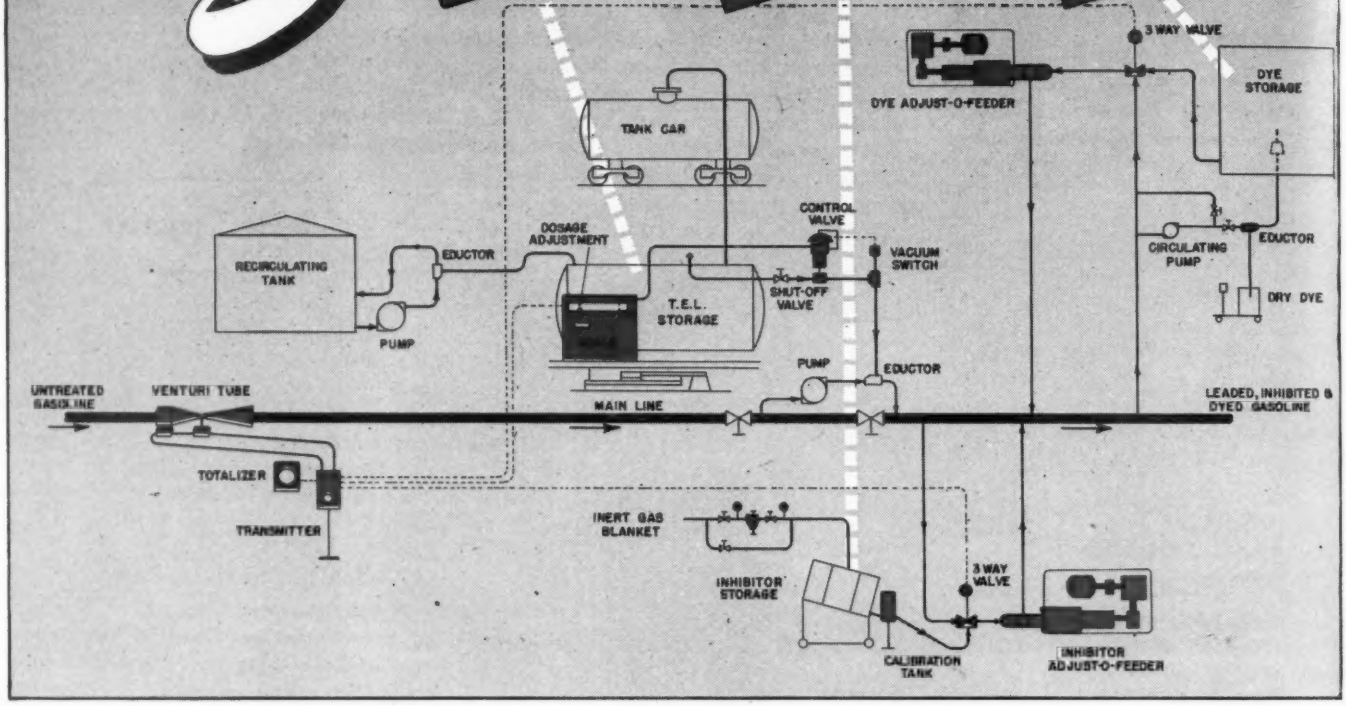
AUTOMATICALLY REGULATED BY GASOLINE FLOW

3 COSTLY ADDITIVES

LEAD

INHIBITOR

DYE



This schematic flowgram illustrates %Proportioneers% Treet-O-Control method of regulating lead, inhibitor and dye injection to a flowing stream of gasoline from a low loss Venturi differential producer.

Additives are handled in concentrated form and are taken directly from the containers in which they are shipped. Transfer of the lead fluid is made from tank car to weigh tank through a vacuum eductor system.

This %Proportioneers% Treet-O-Control system eliminates pre-dilution requiring tankage, piping, circulating pumps and other accessories. It requires minimum space for equipment and assures maximum accuracy in the handling of costly additives.

Ask for further information and recommendations.

% PROPORTIONEERS, INC. %

Write to %PROPORTIONEERS, INC.%, 48 Codding Street, Providence 1, R. I.

Technical service representatives in principal cities of the United States, Canada, Mexico and other Foreign Countries

20—Mass Spectrometer

Isotope-Ratio Mass Spectrometer, Bulletin CE-1803C, a leaflet explaining the operation and application of a new spectrometer designed by Dr. A. O. C. Nier of the University of Minnesota for work involving the use of rare stable isotopes as tracers in the study of reaction mechanisms. Consolidated Engineering Corp.

21—MEK Flammability Limits

Limits of Flammability of Methyl Ethyl Ketone Vapor in Air at Initial Temperatures of 100, 150, *and* 200° *C.,* Bulletin of Research, No. 38, an 18 page report of an investigation of this important petroleum industry chemical by the Underwriters' Laboratories, Inc.

22—Water Treatment

Alkalies and Chlorine in the Treatment of Municipal and Industrial Water, Third Edition, Bulletin No. 8, contains 94 pages devoted to softening, purification, taste, and odor removal, corrosion control, boiler feed water treatment, the use of chemicals and equipment and a list of standard reference works on the subjects covered. Solvay Sales Corp.

23—Fluid Control

Controls for Pressure, Flow, Proportioning of Liquids and Gases, Bulletin 139, 24 pages explaining the operation of instruments designed on the "jet pipe" principle, including basic applications, and instructions on how to calculate an orifice plate. Askania Regulator Co.

24—Engineering Awards

Annual Engineering Undergraduate Award and Scholarship Program of The James F. Lincoln Arc Welding Foundation, a booklet giving the rules and conditions for the next competition, closing date of which is May 15, 1948. J. F. Lincoln Arc Welding Foundation.

25—Industrial Chemicals

Interpretation of Analysis for the Layman, a new edition of a 40-page booklet which emphasizes analytical information on surface active chemicals, including a cross-referenced glossary of terms most frequently used. Nopco Chemical Co.

26—Laboratory Chemicals

Laboratory Chemicals, Catalog No. 247, lists latest prices on gas analysis and other laboratory reagents. Burrell Technical Supply Co.

27—Turnaround Technique

The Kelloggram, Issue No. 5, 1947 Series, is devoted entirely to a picture story of the recent successful turnaround at Gulf Refining Co.'s Philadelphia refinery, which was also presented in PETROLEUM PROCESSING, August, 1947, pgs. 569-574. M. W. Kellogg Co.

28—Steam Specialties

Wright-Austin, Bulletin No. CI 47, a 4-page brochure on steam traps, separators, strainers, and other specialty items which are available for immediate delivery. Wright-Austin Co.

29—Hydrocarbon Enthalpies

Tentative Partial Enthalpies of the Lighter Hydrocarbons, a 14-page reprint of an article under the same title by B. H. Sage, R. H. Olds, and W. N. Lacey, California Institute of Technology, which appeared in the November, 1946, issue of *California Oil World.* Clark Bros. Co., Inc.

30—Oxygen Recorder

Hays Magno-Therm Oxygen Recorder, Advance Bulletin No. 47-844, describes the operation of a new electronic type oxygen recorder the operation of which is based on the magnetic properties of gaseous oxygen. (See "What's New!" October, 1947, page 787, Item 7). Hays Corp.

31—Emulsion Treaters

BS&B Emulsion Treaters, Catalog 54, Part 2, Section 3; 34 pages of description of operation, specifications and complete information on types "F" Stage Treater, "FCW" treater for cold climates, Gas-Free treater, and the "KA" and "KP" systems. Includes prices. Black, Sivalls & Bryson, Inc.

For Your Convenience

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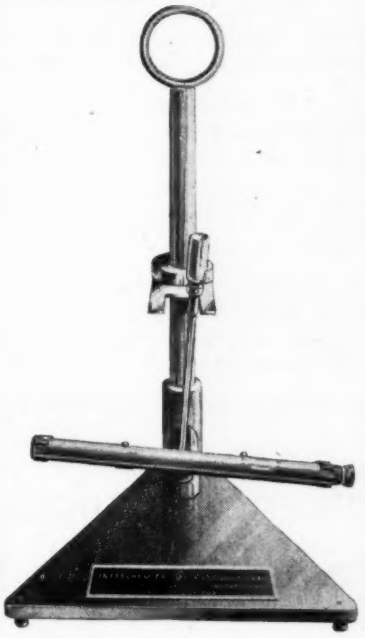

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Insulation, Heat Exchangers, Furnace Tubes Discussed By Mechanical Engineers

Mechanical engineering problems in all phases of the production, transportation and refining of crude petroleum drew the attention of technologists during the Second Annual Petroleum Mechanical Engineering Conference of American Society of Mechanical Engineers at Houston Oct. 6-8.

Of special interest to refiners and natural gasoline plant operators were the mechanical engineering aspects of internally insulated piping and pressure vessels for high-temperature service, in which the insulation of the first commercial hydroforming unit and a Fluid catalyst cracking unit are described. Another paper, which provoked considerable discussion, describes the service life of 18-8 stainless steel furnace tubes and reasons for adopting a policy of discarding them to less rigorous service after 60,000 hours at conditions of 1000 psi and 900-1000° F.

Other papers of particular interest to refiners and natural gasoline manufacturers included:

1—A mathematical discussion of the effect of cross-baffle leakage in a heat exchanger on the estimated heat transfer and the need for writing design specifications with the limitations of fabrication in mind to retain bundle interchangeability and desired thermal characteristics.

2—Application of composite plates (steel plates clad on one or both sides with an alloy), the method of manufacturing, and how they are used in field construction of vessels, such as found in refineries.

3—Accessories for good operation of pressure storage vessels, in which the various types of accessories are described and their applications and limitations discussed.

More than 400 engineers attended the meeting. In general sessions at luncheons and a banquet they were addressed by Gov. Beuford Jester of Texas, on "State Ownership of Lands and Resources Underlying the Marginal Sea;" by Dr. L. W. Blau, Humble Oil & Refining Co. research consultant and chairman of the Engineers Council of Houston; by Dr. E. E. Rosaire, Houston, president of both the National Society of Professional Engineers and the San Jacinto chapter of the state organization; and by E. W. O'Brien, A. S. M. E. president.

Dr. Blau reiterated that engineers "should blow their own horns" and combine with their fellow engineers in attacking mutual problems of a sociological and civic nature instead of splitting into closely-knit groups. He described work on civic and social problems performed by the Engineers Council of Houston; its representation of the various technical societies in the city.

Dr. Rosaire also told engineers they must use their engineering talents on civic and national affairs as well as in workaday jobs. Together with E. W. O'Brien, work of the various engineering groups on the subject of unionization and the questionable status of engineers under previous legislation was described. Both speakers admitted much needs to be done although passage of the Taft-Hartley act settled many of the problems.

Brief reviews of papers having refining and natural gasoline plant applications follow:

Internal Insulation Protects Piping, Vessels in High Temperature Use

"Internally Insulated Piping and Pressure Vessels for High Temperature Service," by P. E. Darling, Pan-American Refining Corp., Texas City, Texas.

APPLICATION of internal insulation to pressure vessels and piping has become an important tool in permitting the utilization of low carbon steel, where the use of bare metal exposed to the high temperature and pressure would either be impractical in cost or impossible from a technical standpoint. The actual application, however, is complex because of the large number of variables which must be considered, including cyclic temperature, fluctuating pressure, corrosion, erosion, and limitations imposed by the characteristics, manufacture, fabrication, erection and maintenance of both insulation and r[illegible].

The design of the first commercial hydroforming unit required either a large number of small diameter reactors or an internally insulated vessel. It was determined that carbon steel could be used providing the metal temperature could be kept to 650° F. or below in vessels and piping. The first lining attempted was with 4.5-in. insulating brick inside and 1.625-in. external block insulation; it began to fail within less than a year. Various repairs were attempted, including 18-8 vapor stops to prevent lining by-passing, over the next three years and finally the search for a better solution to the problem indicated either the installation of a carbon steel or alloy liner or else the use of a suspended refractory tile.

The solution finally developed was the installation of a plastic insulation, 3.5 in. of block insulation and 3 in. of tile in the vessel, which withstood temperature stresses as well as sudden depressurings. Similar problems in the piping finally resulted in the installation of a carbon steel liner. The annular space between liner and pipe is filled with a plastic insulation —"Insulag."

The early experience with the hydroforming unit, although largely negative at the time, led to the development of a successful and economical suspended refractory lining for regenerators in Fluid catalyst units. The dense high-temperature block insulation was laid up very compactly in two layers with all joints pointed in plastic insulation. The refractory tile then was pressed into place in a bed of plastic and positioned to maintain carefully calculated expansion openings. Carbon steel "vapor stops" were provided to prevent by-passing of the liner by catalyst or vapor.

The large spent catalyst lines required, 72-in. I. D., presented several problems which were solved by the use of lapped sectional carbon steel

lines backed by insulating block except at points of extreme turbulence. Longer sections had plastic insulation pumped into the annular space.

Refiner Discards 18-8 Alloy Tube After 60,000 Hours of Service

"Service Life of Austenitic Alloy Furnace Tubes," by Charles S. Pugsley, Jr., Standard Oil Co. of N. J., Louisiana Division, Baton Rouge.

SERVICE life records of 18-8 chrome nickel steel tubes in the radiant section of 1000 psi. thermal cracking unit furnaces are given for an operating period of 16 years. These tubes are 4.5 in. O. D. by 0.4 in. minimum wall, with upset and machined ends. The furnaces operate with normal coil inlet pressures of about 900 to 1000 psig. and temperatures of 900 to 1000° F; the maximum inlet pressure is limited to 1500 psig. by a safety valve and maximum tube metal temperature permitted is 1200° F.

Originally, tubes were inspected carefully almost from the beginning to prevent a service failure; afterwards accurate records were kept on each tube. Five years after initial operation a policy of removing 20 tubes each turnaround was established, replacing them with spares and eventually reinstalling them after reconditioning. As more spares became available, removal for inspection and reconditioning was extended until each tube was removed and reconditioned once each year; finally this was extended to twice annually.

By the end of 1939, after 8 years of operation, there were 125 tubes with over 60,000 service hours each. From the data accumulated it was found that after 50,000 hours the rate of diameter increase, or bulging, was upward; after 60,000 hours external surface roughening was noted and surface defects and cracks became apparent. Only two tubes had failed in service. Therefore a policy of discarding tubes to piping service after 60,000 hours in the furnace was adopted.

In the discussion following presentation, it was brought out that the life of 18-8 tubes is affected by a variety of conditions besides service hours and that discard on this basis alone is undesirable from an economic standpoint. Firing conditions are often a factor, with oil-fired furnaces being tougher on tubes than the gas-fired apparently because of the more uniform heating possible with the latter fuel.

Some service lives of 120,000 hours were reported and one discussor pointed out that, while service failures were prevented, it is doubtful with present-day metals if the full service possibilities of the tubes described had been secured. Trouble due to internal carburization and resulting embrittlement were reported and it was stated that, while laboratory tests did not reveal carburization below 1500° F. temperatures, furnace tubes were found in this condition where temperatures had not exceeded 1200° F. Possibility of a constituent in the topped crude acting as a "carburizing catalyst" was advanced.

Point was also brought out that tubes having small grain-size in the metal as available today, are less likely to give trouble while "sharpshooting" by furnace operators often is responsible for failures.

Mr. Pugsley also commented that during the war, it was necessary to use lower alloys, These had been found so satisfactory that serious consideration now is being given to the use of lower alloys in future tube purchases.

Correcting Heat Losses at Baffles Of Cross-Flow Heat Exchangers

"Effect of Leakage Around Cross-Baffles in a Heat Exchanger," by A. M. Whistler, C. F. Braun & Co., Alhambra, Calif.

IT IS well known that leakage around the baffles of a cross-flow heat exchanger causes losses of efficiency from two sources. First, a loss of temperature difference and, second, a loss of heat transfer rate. The losses from these causes are derived mathematically and, although an exact solution to the problem appears impractical, a method is used which logically is a close approximation to the true losses. In addition to the mathematical derivation, charts are provided for graphical determination of correction for mean temperature difference and comparison of heat transfer rates for different clearances between baffles and different baffle spacings.

It is shown that the loss in temperature difference is less than 5% for the majority of cases. Close approaches in temperature, wide temperature range in shell and few baffles tend to increase the loss. Such a combination can easily require a correction in the mean temperature difference used in design calculations.

The correction for loss in heat transfer rate is comparatively larger. It is worth careful consideration and depends upon clearance between baffles and shell, and baffle spacing. Close spacings require the greatest correction but can be used to advantage provided that allowance is made for decreased efficiency and provided that cleaning still is possible.

In construction, there are several ways of obtaining accuracy in fitting baffles to the shell: (1) bore the shell accurately to size; (2) calibrate by trying progressively smaller discs until one is found which goes through easily and cutting all to this diameter; (3) build shell and element to nominal size, then grind down baffles where they hit; (4) make the baffles accurately to a size which will allow for nominal leakage, having in mind the limitations in fabrication and especially shrinkage due to welding, both at the longitudinal seam and nozzles.

Point is made that often designers of heat exchangers specify tolerances between baffles and shell which are impractical in the shop. The result is that the shop uses one of the first three methods to get the element in, and usually an expensive job results while interchangeability of elements often is entirely lost. Usually the baffles are ground down at the high spots.

Therefore baffle clearance used must be a compromise between efficiency, practical manufacture and maintenance. Specification of impractical clearances is sure to result in either high cost or handfitting which usually results in more clearance than actually was planned in the beginning.

Accessories for Good Operation Of Pressure Storage Vessels

"Accessories for Good Operation of Pressure Storage Vessels," by F. L. Goldsby, Chicago Bridge & Iron Co., Chicago.

ACCESSORIES for pressure storage vessels are divided into accessories for daily operation and those classified as safety devices. Under the first classification are liquid level gages, sampling devices, and temperature and thiefing equipment. The various types of equipment under this classification are described in general terms, and their selection illustrated by means of examples; it is shown that all three are frequently interdependent and selections not capable of standardization.

The basis factors which must be considered in the selection of proper gaging equipment are given as:

1. Operating pressure
2. Frequency with which vessel must be gaged.
3. Purpose of gaging (whether or not gage is used in purchases, sales and transfer of product between companies).
4. Accuracy required.
5. Product handled (hazardous to gager or damaging to certain types of gages).
6. Location of tank with respect to operating office.

In thiefing equipment, besides the interpendence on type of gaging equipment selected, the type of

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PRINCIPLES OF REACTOR DESIGN

By Dr. K. M. Watson & Associates
University of Wisconsin

Containing the complete series of articles which appeared exclusively in PETROLEUM PROCESSING, this booklet is a report of the results of a government-sponsored research program to secure data that could be used in engineering plants for the manufacture of new petroleum-derived chemicals—aromatics for aviation gasoline, toluene, butane-butene for synthetic rubber, and others.

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vessel, operating pressure, quantity and type of sample required, and frequency of sampling must be considered.

Under the second classification—safety devices—the author divides his discussion into venting for pressure relief and vacuum relief, describing the various types of valves, especially for pressure relief.

It is pointed out the ASME and API-ASME Codes provide rules for determining the size of pressure vent openings, but do not codify the determination of maximum amount of vapor to be released through the vent. There usually is no difficult problem to determine what will be necessary to provide for normal atmospheric temperature variations, or for the usual emptying and filling. However, since most users consider additional vents necessary for protection against exposure fire, the big question becomes one of deciding how much vapor will have to be vented to bring pressure down to the desired value.

The only tanks described as not requiring vacuum relief for structural reasons are Hortonspheres rated at working pressures above 82 psi., unit stress of 13,750 psi. and 80% joint efficiency, although they are commonly installed anyway to avoid vapor-locking pumps handling certain types of product. Gas storage spheres seldom require vacuum vents.

To meet safety requirements on construction of pressure storage vessels the following are usually specified:

1. Use steel body fittings below liquid line for all classes of service except where corrosive conditions require special metals.
2. Above liquid line use cast iron or built-up steel fittings up to and including 10 psi.
3. Above liquid line and for working pressures over 10 psi. use steel body fittings except where specifications permit iron body spring-loaded vents rated for higher temperature.
4. Use stainless steel trim on all special fittings except where corrosion conditions require other types of trim.

It is important to keep installation, operating and maintenance instructions up-to-date and in the hands of the men in charge of the storage containers, and not filed in the main office.

Studies Assist in Controlling Contamination in Products Lines

"Interfacial Mixing Characteristics of Products in Products Pipelines," by S. S. Smith and R. K. Schulze, Shell Oil Co., Inc., New York.

THE advancement of the pipeline in the field of transportation of finished petroleum products has paralleled development of techniques for successfully handling many different products through a single line. This type of operation, however, introduces the problem of predetermining and controlling the amount of mixing that results from the movement of one product against another.

Based on laboratory investigation employing a small-scale pipeline, a series of studies was undertaken by Shell to determine the factors governing rate of mixing under various conditions of flow and to develop formulae applicable to designing a commercial size line.

It was finally found that the Reynolds Numbers of the 50/50 mixture of the products involved (as determined by laboratory viscosity determinations) and the length of line traversed (NOT length-to-diameter ratios) could be related to the amount of mixing. This was later confirmed by field tests, but not until recording gravitometers were used as automatic sample determinations in lieu of actual sampling at one-minute intervals. These data have since been converted to plots of Reynolds Numbers of 50/50 mixtures of products and feet of pipe traversed to feet of mixture in the line; they hold regardless of spread of viscosities and diameter of pipe. They also have been plotted as a family of curves each at different Reynolds Number for the 50/50 mixtures and plot distance traveled in the pipeline in feet against the length of the total mixture.

The authors give one caution in using their data in field operations. As nearly as possible the experimental line and pump stations are completely streamlined into a single tube with the absolute possible minimum of side-connections and deadpockets. This is not always strictly true in the field, particularly of older lines built before some of these more critical types of operations were contemplated.

Shell now is using recording gravitometers as a basis for making tender cuts. On its Wood River-Chicago line, where high volatiles like propane are handled on a cycle basis, pressures are kept high enough to assure liquid phase at all times.

Present pump stations along the lines are equipped with sump tanks to receive leakage from stuffing boxes, liquid dropped out of strainers, meters and scrapper traps during normal operations, and sumps are equipped with pumps or eductors to reinject product into the line.

New stations now under construction will have all main line pumps equipped with lubricated mechanical seals, and the sumps will not be expected to handle any continuous drop-out of product. Every effort has been made to eliminate dead pockets, by-pass lines etc., which can add to mixing effects.

Properties, Refinery Applications Of Low Carbon 8½% Ni Steel

"Some Properties of Low Carbon 8½% Nickel Steel" by T. N. Armstrong, Development & Research Division, The International Nickel Co., New York, and G. R. Brophy, Research Laboratory, The International Nickel Co., Bayonne, N. J.

THE increased use of low temperature processing and the advantages associated with storage of gases in liquefied form has stimulated interest in finding a moderately priced construction material resistant to the embrittling effects of low temperatures and which can be fabricated under somewhat the same conditions associated with structural steel.

The following properties of the 8½% Ni samples were studied and reported: impact properties, effect of carbon on impact properties, also manganese and sulfur tensile strength, effect of reheating on strength, fatigue and shear strength, and welding properties. These studies were continued in commercial melts made by several companies interested in the research and resulted in the suggested applications given below.

Low carbon 8½% Ni steel has been produced commercially in the form of billets and bars, plate, seamless tubing and forgings. Flanged and dished heads have been spun and thin gauge strip has been produced by cold rolling. Welded tubing as small as 0.625 in. O.D. by 0.050 in. wall have been made successfully and currently are undergoing tests.

Sound castings with excellent room temperature properties have been made, but the low temperature impact properties have been erratic and in general too low to meet requirements at liquid air temperature. There is as yet no known method for developing full hardness in the case of carburized 8½% Ni steel, as the combination of high carbon and high nickel form austenite that is stable at liquid air temperatures.

While this steel was developed primarily for low-temperature service, and has excellent low temperature (−320° F.) characteristics after normalizing and reheating at 1050°, it is being considered for fields in which low temperature service is not a factor.

The authors caution that the low carbon 8½% Ni steel in no way should be considered as a substitute for the austenitic chromium-nickel stainless steels, although it may be suitable for applications involving a particular type of corrosion; neither is it suggested as a replacement for the low alloy steels for applications involving a particular type of corrosion; neither is it suggested for applications in which high strength is the primary consideration.

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PERSONALS

Petroleum Technologists in the Headlines

Dr. H. R. Snow is plant manager of the Texas City refinery of Pan American Refining Corp., replacing **P. J. Sweeny**, who will devote his full time to the position of first vice president of the refining corporation and of the Mexican Petroleum Corp. of Georgia.

Dr. Snow

Mr. Babin

Dr. Snow is a graduate of the University of Michigan where he also received his master's degree in chemical engineering and his PhD. degree. He joined Standard Oil Co. of Indiana in 1925 and in 1932 was made plant manager of the Neodesha, Kans., refinery. In 1942 he was appointed manager of Rubber Synthetics, Inc., a large butadiene plant which was to be constructed at Gary, Ind., for the government. He went to Texas City from this assignment.

B. F. Babin becomes assistant general superintendent at the Texas City refinery, the position vacated by Dr. Snow. He has been at Texas City since the plant started operations in 1933. He attended Louisiana State University and during the first World War served in the Aviation Corps. He worked for Standard Oil Co. of Louisiana at Baton Rouge and in 1923 joined Mexican Petroleum Corp. of Louisiana at Destrehan. He came to Pan American's Texas City plant in 1933 to direct personnel and safety work and later became foreman and head of the Light Oils department.

P. A. Stewart, formerly assistant department head, succeeds Mr. Babin as head of the Light Oils department.

Also promoted at the Texas City refinery is **W. A. Sullender,** assistant department head, who succeeds **J. B. Hamblen** as head of the Catalytic & Distillation Dept. Mr. Hamblen will head up the manufacturing department of the Destrehan refinery.

R. S. Hatch, formerly manager of the purchasing department of The Texas Co., New York, has been appointed manager of manufacturing and processing for Arabian American Oil Co. A native of Boston, he graduated from Massachusetts Institute of Technology in civil engineering in 1927 and soon went to work for The Texas Co. at Port Arthur, in the engineering department. He held positions in the sales and construction engineering departments and in 1933 returned to Port Arthur as an assistant to the assistant general superintendent of refining. In 1938 he was returned to New York to duty in the purchasing department, becoming assistant manager in 1943 and being promoted to manager in 1946.

* * *

Irving H. Welinsky is a member of the technical staff of the Research & Development Division of Pittsburgh Consolidation Coal Co., assigned to general research. He received his PhD in physical chemistry from New York University in 1938 and for the past 5 years has been with Houdry Process Corp., where he served as head of the process research section. Previously he was with Magnolia Petroleum Co. at Dallas.

Jack F. Chapin is a chemical engineer assigned to operational work on the coal gasification pilot plant. He received a master's degree in chemical engineering from Massachusetts Institute of Technology and was for several years on its teaching staff. He served in the Pacific and Asiatic theatres during the war and was made a lieutenant colonel in Army Ordnance.

William E. Lough has been appointed chemical engineer and will be employed on the coal gasification pilot plant. He is a graduate in chemical engineering from West Virginia University and served in the European theatre during the war, attaining the rank of captain.

* * *

Harold D. Allen and **Carl M. Marberg** are new members of the research staff of Standard Oil Co. (Indiana). They will assist **W. H. Bahkle,** associate director of research, in the company's long range program in petrochemicals. Dr. Allen was formerly with Colgate-Palmolive-Peet Co., in charge of pilot plant and process design work in the division of development engineering. Dr. Marberg was chairman of the organic chemistry research section of the Midwest Research Institute.

N. W. McCrae is superintendent of the Sarnia refinery, succeeding Dr. Huggett. He has been with the Imperial Oil company since 1919 and was on loan during the war to St. Clair Processing Corp., an Imperial Oil subsidiary which was a part of the synthetic rubber program.

* * *

J. F. M. Taylor, formerly president of Shell Development Co. and vice president (manufacturing) Shell Oil Co., Inc., has opened an office in the Russ Bldg., San Francisco, as consultant in petroleum refining and the related fields of research and production of petroleum chemicals.

* * *

Irving E. Lightbown has been made manager of rubber sales for Enjay Co., chemicals marketing affiliate of Standard Oil Co. (New Jersey). He has been with the Jersey interests since 1937 and assistant manager of rubber sales since 1942.

W. V. Rathbone has been made manager of alcohol and chemical sales for Enjay. He has been with Jersey Standard or its affiliates since 1928.

A. Bruce Boehm is manager of Paramine sales. He has been with the company since 1921 and since 1941 has been active in the marketing of the lubricant additives now sold under the brand name Paramine.

* * *

Arthur Highman is manager of the market research department of Oronite Chemical Co., of Standard of California. He graduated in chemical engineering from Illinois Institute of Technology and in 1939 obtained his master's degree from University of Chicago, specializing in marketing and statistics. Later he was with the Navy as a materials engineer and with the Commodities Division on the Army side of the Army and Navy Munitions Board, dealing particularly with chemicals. In 1943 he joined Armour & Co., Chicago, and left in 1946 to take a teaching appointment in Phoenix, Ariz., on the business faculty of the American Institute of Foreign Trade.

Mr. Highman

S. H. Manifold, formerly manager of Oronite's market research department, has been promoted to assistant to the sales manager.

Texas Plant Protects Pumphouse With Independent Foam System

FIRE protection of the gasoline pumphouse at the Port Arthur, Texas, refinery of The Texas Co. has been improved by a new complete foam installation which operates independently of the main plant fire protection system.

A total of 3240 sq. ft. of floor area in the building can be covered with a blanket of foam approximately six inches deep in about 2¾ minutes with the new system, it is reported. This includes the pump room, 72 x 13 ft., and the manifold pit, 72 x 32 ft. A separate motor room is not served by the foam apparatus. The layout is shown in Fig. 1.

The system, designed and supplied by National Foam Systems, Inc., takes its water from the regular 4 in. plant line. The new type of foam solution is fed into the water line by means of a proportioning pump from a storage tank located in one corner of the motor room.

In the pump room at Port Arthur, six overhead spray deflectors distribute the foam so as to cover the tops and sides of all pumps and other machinery, as well as the floor area around the units. Each deflector is capable of producing about 270 gpm. of foam evenly dispersed over a circle approximately 14 ft. in diameter. Being supplied from above, this foam will cling to most vertical surfaces in addition to coating the floor, pump platforms, and the like.

In the manifold pit, the foam is delivered through six floor deflectors, since any fire in this area would be at the level of or under the piping. Each floor deflector introduces foam at the rate of about 540 gpm., and does it simultaneously with the action of the ceiling nozzles in the pump room.

Several methods may be employed to produce the foam solution needed for this type of installation, it is said. At Port Arthur, a storage tank containing 80 gals. of foam liquid, and a rotary pump and motor with a capacity of about 29 gpm. of foam liquid are provided.

The pump takes suction from the storage tank and discharges directly into the water stream—which is at a rate of 454 gpm. at 100 psi. The resulting fire-fighting solution is then piped to the various discharge outlets at the total rate of 483 gpm.

No foam is formed until the solution reaches the discharge nozzles. At that point entrained air, drawn in through an opening in the side of the pipe, forms foam at the ratio of ten gals. to one of solution. The formation of the large volume of foam at the outlets in the distribution system eliminated the necessity for extra large lines.

Operation is manual or, in cases where an attendant is not always on duty, can be made automatic. To start it, the water valve is opened and the foam liquid pump is turned on.

In conjunction with the system at the Port Arthur refinery, there is usually supplied a portable hose line with a discharge nozzle of 600 gpm foam delivery capacity. This nozzle is connected to the water line ahead of the proportioning unit, as shown in Fig. 1, and takes liquid from five gallon cans. It is useful for mopping up purposes or for combatting a small fire which does not require operation of the complete system.

The foam solution used was developed by National Foam from its experience with the U. S. Navy fighting fires during the war.

Five of the six overhead spray deflectors in the pump room at Port Arthur

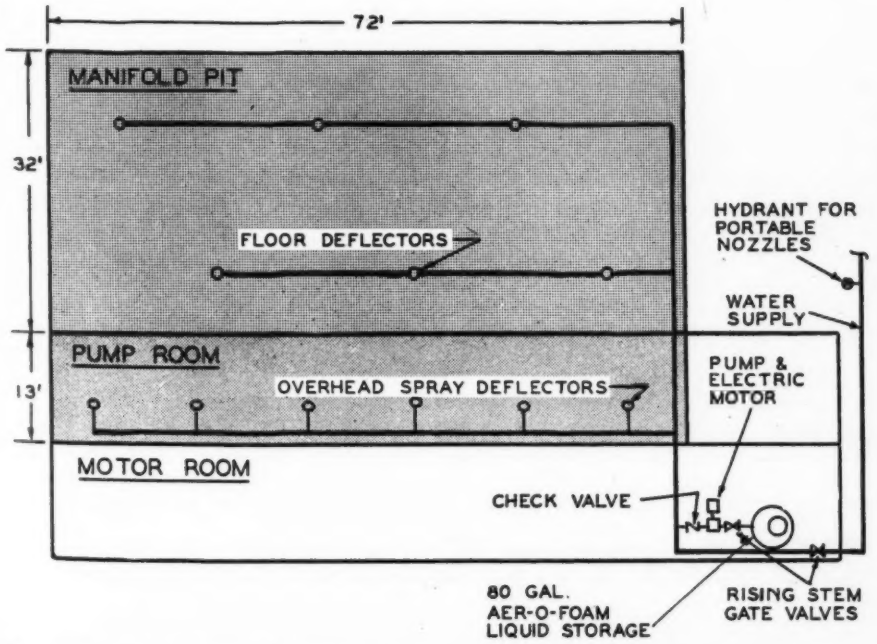
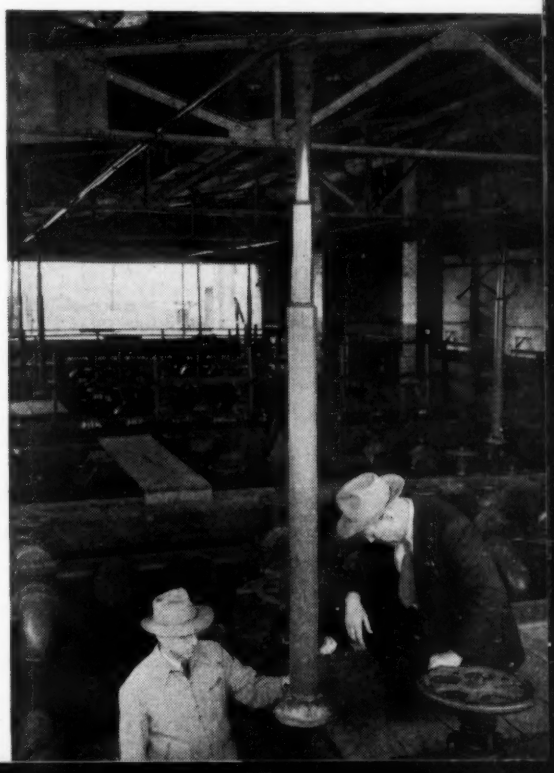


Fig. 1—Layout of foam fire protection system installed in gasoline pumphouse at Port Arthur refinery of The Texas Co. The shaded areas are the ones protected

Company engineers inspect one of the floor deflectors in the foam distribution system in the manifold pit area



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The entire series has now been collected within one cover for easy reference. The book contains all the material as originally published, with the addition of a page of nomenclature and a detailed subject index. Also, the 174 literature references published with the individual articles have been collected into one group as an appendix.

Economic and legislative aspects of the industrial waste problem are covered in the first section, followed by a discussion of the kinds of industrial wastes in general and their effects on waters, plants, and animal life. The balance of the book is devoted pretty much to refinery wastes themselves, and gives details on the investigation of the waste disposal problem and on the treatment of oil-containing wastes. The latter section contains numerous detail drawings on the design and use of the oil-water separator, which the author was instrumental in designing.

The author, W. B. Hart, is well known in the refining industry, having been associated with Atlantic Refining Co., Philadelphia, for over 20 years. He is in charge of Atlantic's progressive waste disposal program and is chairman of the API Committee on Disposal of Refinery Wastes.

With increased attention today being directed at pollution and industrial wastes, it is more important than ever that refinery engineers make certain their plants are not among the offenders. *Industrial Waste Disposal* will be of great value to them, first in understanding the problem of industrial wastes in general, and second in solving it in their specific industry. Even engineers outside the refining industry will find the general treatment of industrial wastes as discussed in the book of value to them.

BIOS Reports Lag in German Chemical Instrumentation

Instrumentation and Control in the German Chemical Industry, by C. H. Gregory, H. B. Appleton, A. P. Lowes, and F. C. Whalen, 5 x 8 in., 157 pages, $6.00

Automatic control and instrumentation in Germany's chemical industry was far behind British or American industry, it is revealed in this new volume. The book is a reprint of a report by a British Intelligence Objectives Sub-Committee (B.I.O.S.), reproduced by permission of the Office of Technical Services, U. S. Dept. of Commerce and published by a private publishing company.

It is a result of a three-month survey during early summer, 1946, on a total of 41 German chemical plants, oil refineries, and instrument manufacturers.

A brief summary statement of results obtained at each target is given first and then followed with detailed descriptions of the type of instruments covered. This latter section is subdivided under the usual instrument classifications.

The investigators found that except for the gigantic I. G. Farbenindustrie, Germany's major chemical firm, and a few of the gasoline plants, instrumentation and automatic control were not used very extensively. However, it was shown that the manufacturers of precision instruments in that country had reached a high stage of development, insofar as accuracy is concerned.

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Manual for Process Engineering Calculations, 1st. Edition, by Loyal Clarke, 6 x 9 in., 448 pages, stiff cloth binding, indexed, $6.00.

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Most process engineers during the course of their work have found it expedient to accumulate a file of useful charts and data sheets of their own devising which are usually made as brief as possible yet complete enough to serve as a ready tool in their work. This manual is that type of information. It consists chiefly of working charts, nomographs, tables, and formulas useful in everyday process engineering problems.

Chapter headings cover such subjects as mathematical data, conversion tables, physical and mechanical properties, thermodynamic data, piping, hydraulics, and flow measurements, heat transfer, combustion, power, pumps, blowers, compressors, distillation, and water.

The author of the manual, Loyal Clarke, is a chemical engineer with the Celanese Corporation of America, and was formerly connected with Petroleum Chemicals, Inc., and the U. S. Bureau of Mines.

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EDITORIAL COMMENTS

WIDENING HORIZONS

Can Catalyst Testing Methods Be Developed for Refiners' Use?

THE Refining Division of the American Petroleum Institute is to be commended for programming for the coming annual API meeting in Chicago a symposium on the testing of cracking catalysts. It is certain that much valuable information on the subject will be presented by the various speakers scheduled. Also, this assembling of information as to what various companies are doing in developing laboratory and pilot plant tests for determining the properties of catalysts used in cracking should stimulate further work, to the end of eventually establishing test methods which can be generally accepted for evaluating the performance of solid catalysts used in petroleum refining.

This entire field of the use of catalytic processes by petroleum refiners is so new that the available information on the subject is as yet very limited. The first extensive use of solid catalysts came with the catalytic processes installed during the war to make aviation gasoline. The Technical Advisory Committee of the Petroleum Industry War Council carried on a good deal of work in testing catalysts to determine their reactivity in cracking operations. Under wartime secrecy its reports were restricted to the company staffs operating the "cat crackers".

Since the end of the war, the large oil companies and a few allied research institutions have expanded their work in the study of catalyst properties, in line with the greater commercial use of this type of refining process, including in addition to cracking, hydroforming, dehydrogenation, polymerization and others. Some of these studies, as will be reported at the API symposium, are already along very advanced lines. They involve the use of x-ray diffraction to determine catalyst surface areas, for example, and also complicated techniques to learn particle size distribution, heat stability, mechanical strength, effect of impurities present and other important characteristics of solid catalysts, as means for predicting performance in actual processing operations. In addition to these laboratory studies, several companies engage in pilot plant work in which the catalyst is tested under conditions simulating as closely as possible commercial processing operations.

Much of the work on catalyst testing now carried on is for the purpose of improving the preparation and manufacture of the catalysts now in commercial use, including the natural, treated and synthetic types of catalysts. However, the information that is being developed is of value to the refiners through showing the relationship of the properties and characteristics of catalysts to their actual performance in processing operations.

These studies on catalysts can be made of even greater value to the industry if eventually methods of laboratory tests, chemical or physical or both, can be set up and generally accepted, which will provide refiners with the means to predict with some degree of accuracy how individual catalysts will perform in their own individual operations. If possible, the test methods should be sufficiently standardized that the results of work in different laboratories will be comparable. The methods arrived at should be such as can be used by the individual refiner without the addition to his laboratory of costly specialized testing equipment.

Such a program need not affect the present advanced studies of catalyst reactivity under way in many of the larger laboratories. As now carried on, these individual programs will be of great value to the industry in general through the development of a relatively new field of research in petroleum refining. Along with this work, however, should be kept in mind the need for providing relatively simple methods and techniques which individual refiners can use in evaluating the performance of catalysts for use under their own operating conditions.

A Lot of Dirt to Scratch Up

THE Bureau of Mines technologists devoting their time to the Synthetic Liquid Fuels program have much to say about the vast oil reserves contained in the shale deposits of Colorado and other sections of the country.

Even more impressive, however, as far as figures go, is the data on the volume of shale that must be handled and processed to recover the oil in it, and we don't hear much about this. The question comes up as to whether any commercial operations that might by any stretch of the imagination be considered economically practicable, could provide for the recovery of any volume of oil from shale in our own or our children's times, to say nothing of recovering the oil at the rate needed in an emergency.

Data issued by the Bureau of Mines at the dedication of the oil-shale demonstration plant at Rifle, Col., May 17 refers to a supply in Naval Oil-Shale Reserve No. 1, in western Colorado of 15 billion barrels, equivalent to three-fourths of our known petroleum reserves. While the oil content of the shale varies widely, from 10 to 60 gallons to the ton, for the sake of easy figuring let's assume an average yield of 21 gallons per ton. That would mean that 30 billion tons of shale would have to be mined, crushed, transported to an extraction plant, processed and the spent solid material removed to recover all this oil. Try to imagine the equipment required, and the cost of its investment, to handle the tremendous volume of solid material needed to produce any great volume of oil from shale.

The same source says that two N-T-U batch type retorts each capable of holding 40 tons of raw oil shale, are being installed in the processing plant. We aren't told how much time the batch retorting operation consumes. However, assume a commercial plant capacity 100 times daily that of the present experimental single retort, which would seem to call for a tidy investment, to process 4000 tons a day. The yield of raw oil from the operation, again using an average 21 gallons per ton yield, would be 2000 barrels, an exceedingly small output as petroleum refiners figure plant capacity.

The possible yield from the oil shale reserves in continental U.S. may be at least 50 times the present annual consumption of petroleum, as the Mines Bureau technologists point out. A more important point is that the mechanical and economic problems involved in moving great volumes of the solid material would seem to make it impossible to recover the oil at a rate that would be important when we may need it. This is the phase of the problem to which the Bureau should be devoting its time, rather than to methods for processing the shale to recover the oil contained in it.

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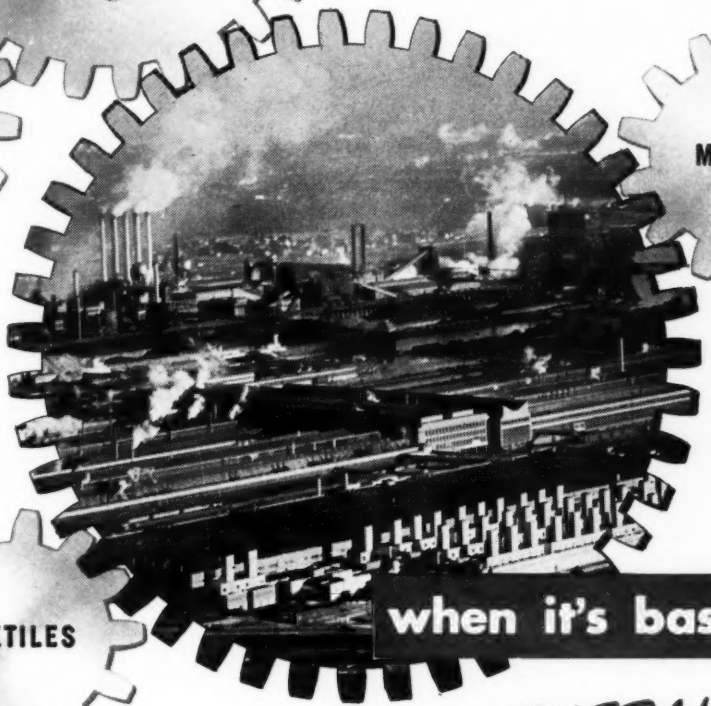

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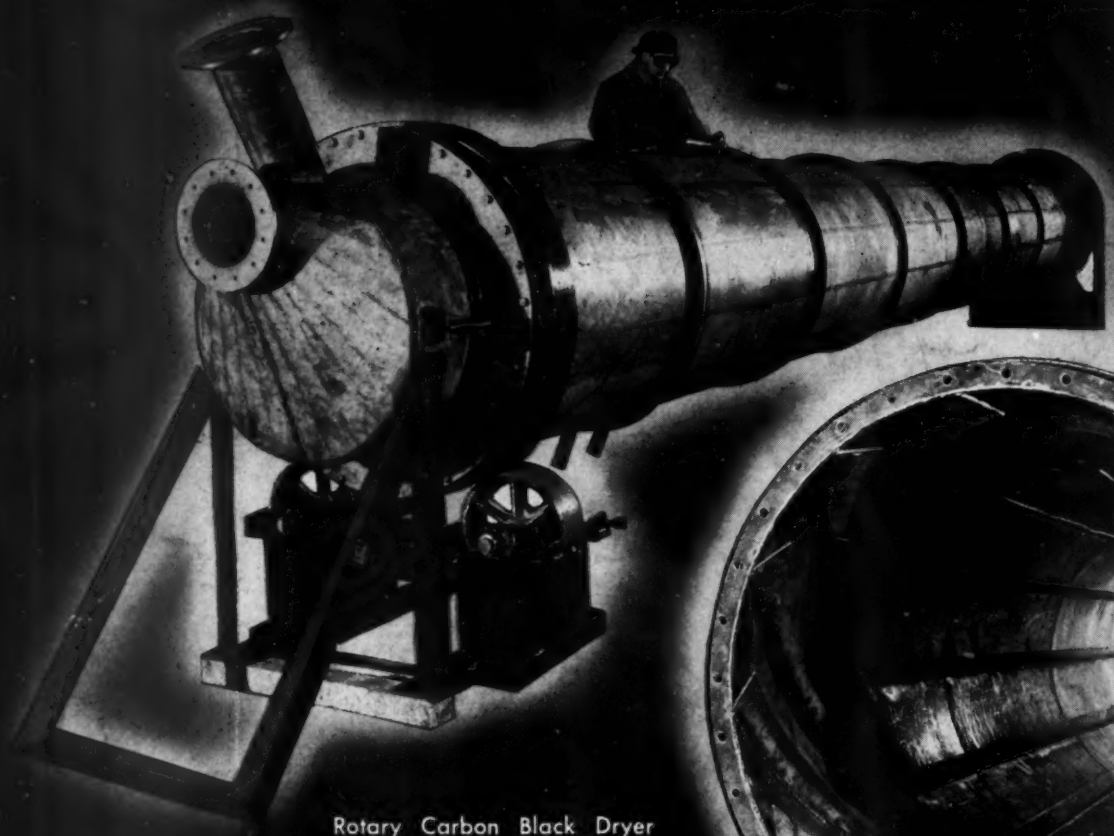
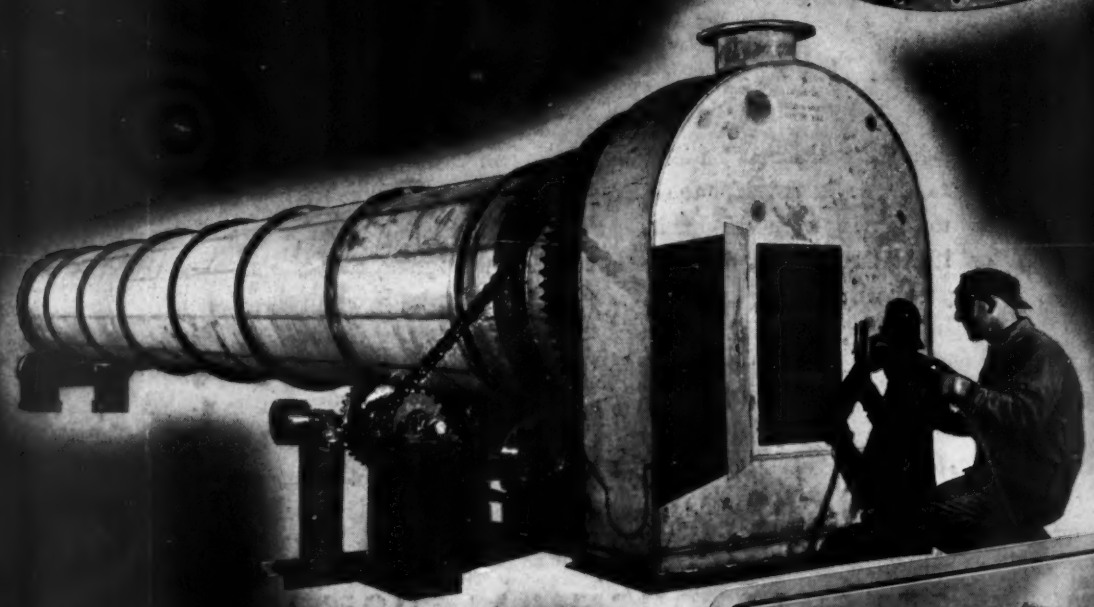


Rotary Carbon Black Dryer fabricated at Wyatt's from solid stainless steel type 310.



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